

08/776,528

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* U. S. P A T E N T T E X T F I L E *

* * * * *

L1 11717 OXIME?

182380 ETHYLENE

146336 CARBONATE

=> s propylene (w) carbonate

118302 PROPYLENE

146336 CARBONATE

=> s 12 or 13

L4 5822 L2 OR L3

=> s 11 (3P) 14

L5 29 L1 (3P) L4

=> d 15 1-29 ab bib hit

L5: 1 of 29

Electrolyte compositions in which a salt is disposed in a thiol-ene matrix. The compositions retain their shape under operating conditions and exhibit an ionic conductivity of at least 1×10^{-6} when measured in the absence of solvent at 25.degree. C.

L5: 1 of 29

DATE ISSUED: Jan. 14, 1997

TITLE: Polymer electrolyte composition based upon thiol-ene

chemistry

INVENTOR: Kejian Chen, Woodbury, MN
Haitao Huang, Woodbury, MN
ASSIGNEE: Minnesota Mining and Manufacturing Company, St. Paul, MN
(U.S. corp.)
APPL-NO: 08/435,110
DATE FILED: May 1, 1995
ART-UNIT: 111
PRIM-EXMR: Stephen Kalafut
ASST-EXMR: Carol Chaney
LEGAL-REP: Gary L. Griswold, Walter N. Kirn, Scott A. Bardell

DETDESC:

DETD(20)

The amount of photoinitiator generally ranges from about 0.01 to about 10 parts per 100 parts of reactants. Examples of suitable polymerization initiators include benzoin ethers, substituted benzoin ethers such as benzoin methyl ether or benzoin isopropyl ether, substituted acetophenones such as 2,2-diethoxy-acetophenone, and 2,2-dimethoxy-2-phenyl-acetophenone, substituted alpha-ketols such as 2-methyl-2-hydroxypropiophenone, aromatic sulphonyl chlorides such as 2-naphthalene sulphonyl chloride, and photoactive ****oximes**** such as 1-phenyl-1,1-propanedione-2-(O-ethoxycarbonyl) ****oximes****. Other suitable initiators include 2,4-bistrichloro-methyl-6-substituted-s-triazines, and benzophenone with an amine (for example and p-N,N-diethylamino) ethyl benzoate). Sensitizers such as phenanthrene may be used in conjunction with the photoinitiators as well.

DETDESC:

DETD(22)

Following polymerization, the electrolyte compositions may be swollen (if desired) with solvent. Aprotic liquids are preferred. Such liquids are generally dry (e.g., have a water content less than about 100 ppm, preferably less than about 50 ppm). Examples of suitable aprotic liquids include linear ethers such as diethyl ether and 1,2-dimethoxyethane; cyclic ethers such as tetrahydrofuran, 2-methyltetrahydrofuran, dioxane, dioxolane, and 4-methyldioxolane; esters such as methyl formate, ethyl formate, methyl acetate, diethyl carbonate, ****propylene** carbonate****, ****ethylene** carbonate****, and butyrolactones; nitriles such as acetonitrile and benzonitrile; nitro compounds such as nitromethane or nitrobenzene; amides such as N,N-dimethylformamide, N,N-diethylformamide, and N-methylpyrrolidone; sulfoxides such as dimethyl sulfoxide; sulfones such as dimethylsulfone, tetramethylene sulfone, and other sulfolanes;

oxazolidones such as N-methyl-2-oxazolidone; and mixtures thereof.

US PAT NO: 5,591,354 [IMAGE AVAILABLE]

L5: 2 of 29

ABSTRACT:

There is disclosed a process of etching the surface of polymeric materials made of polymers having at least one oxidatively degradable functionality which comprises treating said surface with a solution of at least one member of the etchant group consisting of nitronium, nitrosonium ions and complexes thereof, in a solvent containing less than 40% by weight of water and nitroacidium ions and complexes thereof. The process further comprises use of cosolvents and swellants. It also includes pre and post etching steps directed to the preparation of the surface for etching and removal of the etchant respectively.

US PAT NO: 5,591,354 [IMAGE AVAILABLE]

L5: 2 of 29

DATE ISSUED: Jan. 7, 1997

TITLE: Etching plastics with nitrosyls

INVENTOR: Gordhanbhai N. Patel, Somerset, NJ
Subhash H. Patel, Hoboken, NJ

ASSIGNEE: JP Laboratories, Inc., Middlesex, NJ (U.S. corp.)

APPL-NO: 08/327,237

DATE FILED: Oct. 21, 1994

ART-UNIT: 119

PRIM-EXMR: Thi Dang

LEGAL-REP: Omri M. Behr, Esq.

SUMMARY:

BSUM(7)

Prior to etching plastics for electroless plating, they are sometimes pre-treated with a solvent system to swell the surfaces. Solvent pretreated/swollen surfaces are micro-toughened and hence are relatively easy to etch and hence provide better adhesion of the plated metals. A number of solvents, such as butyrolactone, **propylene** **carbonate**, glymes, and methanol, have been proposed in a number of patents (e.g., U.S. Pat. Nos. 3,445,350; 3,689,303; 3,769,061; 3,795,622; 3,963,590; 4,063,004; 4,131,698; 4,775,449; 5,019,425; 5,132,191; 5,178,956; 5,180,639; and 5,185,185). However, in order to get improved adhesion, the solvent must be removed from the surface and the surface still requires etching. There is a need for an etching formulation which simultaneously swells the surface.

SUMMARY:

BSUM(9)

NSA is known to react as a nitrosyl cation or nitrosonium cation ($\text{NO}^{\text{sup.}}$) with other nucleophiles including unsaturated compounds, such as 1,3-dienes [Chem. Ber. 99, 556 (1966)]. $\text{NO}^{\text{sup.}}$ is also capable of halogen abstraction from alkyl halides [G. A. Olah et al, Synthesis, 274 (1979) and 713 (1983)]. $\text{NO}^{\text{sup.}}$ is a mild oxidizing agent. It is a powerful one electron oxidant [Lee et al, Inorg. Chem., 29, 4196 (1990) and Kim et al, J. Amer. Chem. Soc., 113, 4962 (1991)] and is therefore used as an oxidative dopant for polymers. It has been used to oxidatively cleave **oximes**, hydrazones, thioketals, ethers etc. to their corresponding carbonyl compounds [Synthesis, 418 (1977), 273 (1979), 609 and 610 (1976), J. Org. Chem., 42, 309 (1977)]. The $\text{NO}^{\text{sup.}}$ is electrophilic, and therefore reacts with a large number of basic molecules. The nitrosonium cation also forms adducts with crown ethers. Though the reactions of NSA have been reported with a number of small molecules, there is no report on its reactions with polymers, especially for etching and chemical modification of the polymers.

US PAT NO: 5,582,693 [IMAGE AVAILABLE]

L5: 3 of 29

ABSTRACT:

3-Carene and limonene cannot be separated from each other by rectification because of the closeness of their boiling points. They are readily separated by azeotropic distillation. Effective agents are: cyclopentanol, 2-nitropropane, ethyl formate, amyl acetate, dimethyl carbonate, tetrahydrofuran, acetic acid and 2-amino-2-methyl-1-propanol.

US PAT NO: 5,582,693 [IMAGE AVAILABLE]

L5: 3 of 29

DATE ISSUED: Dec. 10, 1996

TITLE: Separation of 3-carene and limonene by azeotropic distillation

INVENTOR: Lloyd Berg, 1314 S. Third Ave., Bozeman, MT 59715

ASSIGNEE: Lloyd Berg, Bozeman, MT (U.S. indiv.)

APPL-NO: 08/584,983

DATE FILED: Jan. 11, 1996

ART-UNIT: 132

PRIM-EXMR: Virginia Manoharan

SUMMARY:

BSUM(17)

Table 4 lists the compounds that are effective in separating 3-carene from limonene. They are amyl acetate, 1-butanol, anisole, isopropyl ether, dioxolane, ethyl formate, methyl acetate, methyl propionate,

methyl formate, ethyl acetate, propyl propionate, n-butyl propionate, dimethyl carbonate, t-amyl alcohol, 3-pentanone, 2-octanone, 2-pentanone, 3-methyl-2-butanone, 2,3-butanedione, cyclohexanone, acetonitrile, butyronitrile, 2-butanone, diethylene glycol methyl ether, butyl amine, dipropyl amine, triethyl amine, 2-methoxyethanol, acetone, p-cresol, pyridine, tetrahydrofuran, 2,6-dimethyl phenol, **propylene**
 carbonate, 2-methyl-2,4-pentanediol, 1,1,3,3-tetramethyl urea, 4-hydroxy-4-methyl-2-pentanone, methyl ethyl ketoxime, 2-nitropropane, butyraldehyde **oxime**, 1,2-Methylenedioxybenzene, 1-methoxy-2-propanol, 2-amino-2-methyl-1-propanol, 2-butoxyethanol, propoxypropanol, 4-fluoro benzaldehyde and cyclopentanol.

SUMMARY:

BSUM(18)

Table 5 lists the compounds that are effective in separating phellandrene from 3-carene and limonene. They are dimethylformamide, ethyl lactate, cyclopentanol, 4-hydroxy-4-methyl-2-pentanone, formic acid, butyraldehyde **oxime**, 1,2-methylene dioxybenzene, 1-methoxy-2-propanol, 2-dimethylamine-2-methyl-2-propanol and 2-nitropropane.

SUMMARY:

BSUM(21)

TABLE 4

Effective Azeotropic Distillation Agents For Separating 3-Carene From Limonene

Agent	Temp., .degree.C.	Rel. Vol. 3-Car/Lim
Amyl acetate	153	1.65
1-Butanol	120	1.55
Anisole	152	1.4
Isopropyl ether	91	1.45
Dioxolane	78	1.4
Ethyl formate	58	1.7
Methyl acetate	56	1.65
Methyl propionate	83	1.35
Methyl formate	31	1.75
Ethyl acetate	83	1.3
Propyl propionate	138	1.5
n-Butyl propionate	133	1.3

Dimethyl carbonate	92	1.3
t-Amyl alcohol	107	1.35
3-Pentanone	119	1.35
2-Octanone	162	1.35
3-Methyl-2-butanone		
	104	1.35
2,3-Butanedione	93	1.45
2-Pentanone	110	1.4
Cyclohexanone	150	1.3
Acetonitrile	80	1.5
Butyronitrile	117	1.7
2-Butanone	90	1.35
Diethylene glycol methyl ether		
	165	1.3
Butyl amine	86	1.4
Dipropyl amine	119	1.3
Triethyl amine	99	1.3
2-Methoxyethanol	120	1.4
Acetone	56	1.5
p-Cresol	156	1.4
Pyridine	114	1.5
Tetrahydrofuran	88	1.4
2,6-Dimethyl phenol		
	146	1.4
2-Methyl-2,4-pentanediol		
	166	1.3
1,1,3,3-Tetramethyl urea		
	163	1.35
Propylene **carbonate**		
	166	1.3
4-Hydroxy-4-methyl-2-pentanone		
	131	1.55
Methyl ethyl ketoxime		
	145	1.45
Cyclopentanol	127	1.6
Butyraldehyde **oxime**		
	142	1.4
1,2-Methylene dioxybenzene		
	164	1.3
1-Methoxy-2-propanol		
	120	1.35
2-Amino-2-methyl-1-propanol		
	147	1.5
2-Nitropropane	120	1.55
2-Butoxyethanol	160	1.3
Propoxypropanol	149	1.5
4-Fluoro benzaldehyde		

SUMMARY:

BSUM(22)

TABLE 5

Effective Azeotropic Distillation Agents For Separating
Phellandrene From 3-Carene and Limonene

Agent	Temp. .degree.C.	Rel. Vol.
		Lim/Phl
Dimethylformamide	127	1.6
Ethyl lactate	146	1.45
Cyclopentanol	137	2.0
4-Hydroxy-4-methyl-2-pentanone		
	148	1.45
Formic acid	103	1.3
Butyraldehyde **oxime**	145	1.35
1,2-Methylene dioxybenzene		
	164	1.35
1-Methoxy-2-propanol	126	1.45
2-Dimethylamine-2-methyl-2-propanol		
	150	1.8
2-Nitropropane	120	1.5

US PAT NO: 5,523,377 [IMAGE AVAILABLE]

L5: 4 of 29

ABSTRACT:

Blocked (cyclo)aliphatic polyisocyanates wherein 30 to 70 equivalent-% of the isocyanate groups are blocked with butanone oxime, 30 to 70 equivalent-% of the isocyanate groups are blocked with 1,2,4-triazole and 0 to 30 equivalent-% of the isocyanate groups are blocked with other blocking agents; a method for their preparation by blocking lacquer polyisocyanates with these blocking agents, optionally with the prior, simultaneous or subsequent modification of the polyisocyanates with hydrazine derivatives containing one or more isocyanate-reactive groups; and the use of the blocked polyisocyanates as cross-linking agents for organic polyhydroxy compounds in one-component polyurethane stoving compositions.

US PAT NO: 5,523,377 [IMAGE AVAILABLE]

L5: 4 of 29

DATE ISSUED: Jun. 4, 1996
TITLE: Blocked polyisocyanates
INVENTOR: Eberhard Konig, Leverkusen, Federal Republic of Germany
Theodor Engbert, Koln, Federal Republic of Germany
Thomas Klimmasch, Leverkusen, Federal Republic of Germany
Manfred Bock, Leverkusen, Federal Republic of Germany
ASSIGNEE: Bayer Aktiengesellschaft, Leverkusen, Federal Republic of
Germany (foreign corp.)
APPL-NO: 08/336,029
DATE FILED: Nov. 8, 1994
ART-UNIT: 127
PRIM-EXMR: Rachel Johnson
LEGAL-REP: Joseph C. Gil, Thomas W. Roy

DETDESC:

DETD(8)

Butanone **oxime** is used as blocking agent A) and 1,2,4-triazole is used as blocking agent B). Other optional blocking agents include secondary amines such as diisopropylamine or imidazole, CH-acid compounds such as diethyl malonate, and .epsilon.-caprolactam.

DETDESC:

DETD(11)

The hydrazine adducts can be obtained by reacting 1 mole of hydrazine hydrate with 2 moles of a cyclic carbonate such as **ethylene** **carbonate**, isopropylene carbonate or 1,3-neopentyl carbonate, e.g., in boiling toluene. The water in the hydrate is removed by azeotropic distillation.

DETDESC:

DETD(30)

A commercially available polyisocyanate blocked only with butanone **oxime**, prepared from the lacquer polyisocyanate containing isocyanurate groups described in Example 1, and present as a 65% solution in Solvesso 100 solvent. The NCO-equivalent weight of the solution (based on the blocked isocyanate groups) was 518. The viscosity of the solution (23.degree. C.) was 14,000 mPa.s.

DETDESC:

DETD(32)

This example differs from Example 1 due to the additional incorporation of a hydrazine adduct prepared from 1 mole of hydrazine hydrate and 2 moles of **propylene** **carbonate** and having a molecular weight of 236 and corresponding to the structural formula ##STR5##

DETDESC:

DETD(33)

Charge:

792	g	(2.2 g. equiv.)	of the lacquer polyisocyanate containing isocyanurate groups described in Example 1
69	g	(1.0 g. equiv.)	1,2,4-triazole
87	g	(1.0 g. equiv.)	butanone **oxime**
23.6	g	(0.2 g. equiv.)	of the above hydrazine adduct
253.4	g		methoxypropyl acetate

DETDESC:

DETD(35)

The polyisocyanate and methoxypropyl acetate were mixed together and crystalline flakes of 1,2,4-triazole (Fp. 120.degree. C.) were added to the stirred solution. The mixture was heated to 100.degree. C. in stages, during which time the 1,2,4-triazole completely dissolved. The melted hydrazine adduct was then added in one pouring and reacted for 1 to 2 hours at 100.degree. C. until the NCO-content had fallen to approximately 3.8; the calculated value was 3.7. The reaction mixture was then cooled to 50.degree. C. and butanone **oxime** was added in a manner such that the temperature did not rise above 70.degree. C. After reaction for a further hour, additional small quantities of butanone **oxime** were added until no NCO groups were detectable by IR spectroscopy.

DETDESC:

DETD(38)

Charge:

587.0 g

(1.63 g. equiv.)

of the lacquer polyisocyanate containing
isocyanurate groups described in
Example 1

200.0 g

(1.0 g. equiv.)

of a commercially available lacquer
polyisocyanate containing isocyanurate
groups, prepared from 1,6-diisocyanato-
hexane and having an NCO-content of
about .21% and a viscosity at 23.degree. C. of
about 3,000 mPa .multidot. s.

79.3 g

(1.15 g. equiv.)

1,2,4-triazole

100.0 g

(1.15 g. equiv.)

butanone **oxime**

39.0 g

(0.33 g. equiv.)

of the hydrazine-**propylene** **carbonate**
adduct described in Example 3

383.7 g

methoxypropyl acetate

DETDESC:

DETD(40)

The two polyisocyanates and the methoxypropyl acetate were mixed together and crystalline flakes of 1,2,4-triazole were added to the solution with stirring. The temperature was elevated to 100.degree. C. during which time the triazole (Fp. 120.degree. C.) dissolved. The reaction was continued at 100.degree. C. for 1 hour. The melted hydrazine adduct was then added in one pouring and the reaction continued for a further 2 hours at 100.degree. C., until an NCO-content of 3.8% was attained; the calculated value was 3.75%. The reaction mixture was cooled to 50.degree. C. and butanone **oxime** was added in a manner such that the temperature did not exceed 70.degree. C. Stirring was continued at 70.degree. C. for approximately 1 hour until no further NCO-groups were detectable by IR spectroscopy and the mixture was then decanted.

ABSTRACT:

The present invention relates to process for preparing a coated substrate having improved resistance to yellowing which comprises coating a substrate with a one-component coating composition containing

a) a blocked polyisocyanate which is the reaction product of a polyisocyanate with an oxime or lactam blocking agent for isocyanate groups and

b) a compound containing at least two isocyanate-reactive groups, provided that component a) and/or component b) contains the group, --CO--(R)N--N(R)--CO--, wherein R represents hydrogen or an optionally substituted hydrocarbon radical, in an amount of 0.01 to 5% by weight, based on the solids content of components a) and b),

by applying the one-component coating composition wet-on-wet to a coating composition containing acid groups and/or melamine resins which has previously been applied to the substrate or by adding a melamine resin to the one-component coating composition prior to applying it to the substrate, which may optionally have been precoated.

The present invention also relates to the coated substrate having improved resistance to yellowing prepared by this process.

US PAT NO: 5,510,443 [IMAGE AVAILABLE] L5: 5 of 29
DATE ISSUED: Apr. 23, 1996
TITLE: Process for preparing a coating with improved resistance
to yellowing and the resulting coating
INVENTOR: Myron W. Shaffer, Coraopolis, PA
Terry A. Potter, Beaver, PA
Lanny D. Venham, Paden City, WV
Peter D. Schmitt, Glen Dale, WV
ASSIGNEE: Bayer Corporation, Pittsburgh, PA (U.S. corp.)
APPL-NO: 08/031,423
DATE FILED: Mar. 15, 1993
ART-UNIT: 127
PRIM-EXMR: Nathan M. Nutter
ASST-EXMR: John M. Cooney, Jr.
LEGAL-REP: Joseph C. Gil, Thomas W. Roy

DETDDESC:

DETD(57)

A round bottom flask was charged with 223.2 g of hydrazine hydrate (64% hydrazine by weight), 223.2 g of 2-propanol, and 223.2 g of toluene. To this mixture were added 500.0 g of **propylene** **carbonate** in three portions over a one hour time period. The temperature was not allowed to exceed 80.degree. C. After stirring this mixture for one hour, the

temperature was maintained at 80.degree. C. and the excess solvent and water was removed under reduced pressure using a water aspirator. After one hour the temperature was raised to 100.degree. C. and a full vacuum was used to remove the remaining solvent and water. After about 1.5 hours, the final product was a clear light yellow viscous liquid.

DETDESC:

DETD(59)

514.2 grams of Polyisocyanate V and 260 grams of Aromatic 100 solvent were added to a three neck, two liter, round bottom flask equipped with a mechanical stirrer and thermocouple. This mixture was stirred until homogeneous and then 208 grams of butanone ****oxime**** were added dropwise at a rate which allowed the exothermic reaction to keep the solution temperature at 50.degree. C. When all of the butanone ****oxime**** had been added, the mixture was kept at 50.degree.-60.degree. C. for one additional hour. The isocyanate content was determined to be 1.1%. Next, 20 grams of Additive A were added and the mixture was heated to 80.degree. C. to dissolve this powder. The solution was kept at 80.degree. C. for two hours. At this time the isocyanate content was determined to be essentially zero by IR spectrum. 3.13 equivalent percent of the isocyanate groups of the polyisocyanate were modified with the anti-yellowing compound, i.e., Additive A.

US PAT NO: 5,504,178 [IMAGE AVAILABLE]

L5: 6 of 29

ABSTRACT:

The present invention relates to a blocked polyisocyanate which is the reaction product of a polyisocyanate with

- i) 60 to 99.9 equivalent percent, based on the equivalents of isocyanate groups of the polyisocyanate prior to blocking, of an oxime or lactam blocking agent for isocyanate groups and
- ii) 0.1 to 40 equivalent percent, based on the equivalents of isocyanate groups of the polyisocyanate prior to blocking, of hydrazine and/or a compound which is soluble in either the polyisocyanate, the blocking agent or an optional organic solvent for the polyisocyanate and contains the group, $H(R)N--NR--CO--$, wherein the carbonyl group is bound to carbon or nitrogen and R represents hydrogen or an optionally substituted hydrocarbon radical. The present invention also relates to a one-component coating composition containing this blocked polyisocyanate and a compound containing at least two isocyanate-reactive groups and to coated substrates prepared from this one-component coating composition which additionally contains an acid.

US PAT NO: 5,504,178 [IMAGE AVAILABLE] L5: 6 of 29
DATE ISSUED: Apr. 2, 1996
TITLE: One-component coating compositions containing oxime- or
lactam-blocked polyisocyanates which have improved
resistance to yellowing
INVENTOR: Myron W. Shaffer, Coraopolis, PA
Terry A. Potter, Beaver, PA
Lanny D. Venham, Paden City, WV
Peter D. Schmitt, Glen Dale, WV
ASSIGNEE: Bayer Corporation, Pittsburgh, PA (U.S. corp.)
APPL-NO: 08/459,936
DATE FILED: Jun. 2, 1995
ART-UNIT: 127
PRIM-EXMR: James J. Seidleck
ASST-EXMR: R. F. Johnson
LEGAL-REP: Joseph C. Gil, Thomas W. Roy

DETDESC:

DETD(19)

A round bottom flask was charged with 223.2 g of hydrazine hydrate (64% hydrazine by weight), 223.2 g of 2-propanol, and 223.2 g of toluene. To this mixture were added 500.0 g of **propylene** **carbonate** in three portions over a one hour time period. The temperature was not allowed to exceed 80.degree. C. After stirring this mixture for one hour, the temperature was maintained at 80.degree. C. and the excess solvent and water was removed under reduced pressure using a water aspirator. After one hour the temperature was raised to 100.degree. C. and a full vacuum was used to remove the remaining solvent and water. After about 1.5 hours, the final product was a clear light yellow viscous liquid.

DETDESC:

DETD(21)

514.2 grams of Polyisocyanate V and 260 grams of Aromatic 100 solvent were added to a three neck, two round bottom flask equipped with a mechanical stirrer and thermocouple. This mixture was stirred until homogeneous and then 208 grams of butanone **oxime** were added dropwise at a rate which allowed the exothermic reaction to keep the solution temperature at 50.degree. C. When all of the butanone **oxime** had been added, the mixture was kept at 50.degree.-60.degree. C. for one additional hour. The isocyanate content was determined to be 1.1%. Next, 20 grams of Additive A were added and the mixture was heated to 80.degree. C. to dissolve this powder. The solution was kept at 80.degree. C. for two hours. At this time the isocyanate content was

determined to be essentially zero by IR spectrum. 3.13 equivalent percent of the isocyanate groups of the polyisocyanate were modified with the anti-yellowing compound, i.e., Additive A.

US PAT NO: 5,434,306 [IMAGE AVAILABLE]

L5: 7 of 29

ABSTRACT:

The invention relates to a process for the hydroxyalkylation of aldoximes and ketoximes by reacting said ****oximes**** with unsubstituted or substituted ethylene or ****propylene**** ****carbonate**** in the presence of a catalyst, which comprises the use of catalytic amounts of a N-alkylated, stable, organic amidine base or a pyridine which is substituted by a secondary amino group. The compounds are important intermediates for the synthesis of herbicides.

US PAT NO: 5,434,306 [IMAGE AVAILABLE]

L5: 7 of 29

DATE ISSUED: Jul. 18, 1995

TITLE: Process for the preparation of O-substituted oximes

INVENTOR: Denis Blaser, Monthey, Switzerland

ASSIGNEE: Ciba-Geigy Corporation, Ardsley, NY (U.S. corp.)

APPL-NO: 08/333,910

DATE FILED: Nov. 3, 1994

ART-UNIT: 129

PRIM-EXMR: Peter O'Sullivan

LEGAL-REP: Marla Mathias

ABSTRACT:

The invention relates to a process for the hydroxyalkylation of aldoximes and ketoximes by reacting said ****oximes**** with unsubstituted or substituted ethylene or ****propylene**** ****carbonate**** in the presence of a catalyst, which comprises the use of catalytic amounts of a N-alkylated, stable, organic amidine base or a pyridine which is substituted by a secondary amino group. The compounds are important intermediates for the synthesis of herbicides.

SUMMARY:

BSUM(1)

The present invention relates to a process for the preparation of O- ω -hydroxyalkyloximes by reacting ****oximes**** with alkylene carbonates in the presence of catalytic amounts of an amidine or a pyridine base.

SUMMARY:

BSUM(2)

These ****oximes**** are important intermediates for herbicides, inter alia those disclosed in U.S. Pat. Nos. 4,545,807 and 4,687,849.

SUMMARY:

BSUM(3)

U.S. Pat. No. 4,687,849 relates to a process for the preparation of 2-[(isopropylideneamino)oxy]ethanol, which comprises reacting acetone ****oxime**** in aqueous medium, in the presence of catalytic mounts of Ca(OH)_2 , with ethylene oxide. One drawback of this process is that ethylene oxide is difficult to produce on an industrial scale and can only be handled with stringent safety regulations, as it is highly explosive and extremely poisonous. Another drawback is that the process results in substantial amounts of wastewater that have to be purified. It is also already known that 2-[(isopropylideneamino)oxy]ethanol can be prepared by reacting acetone ****oxime**** with ****ethylene** carbonate****. Potassium fluoride and tetramethylammonium bromide can be used as catalysts for this reaction. This process is described, inter alia, in R. Klauser et al. in ACS Symposium Ser. 1991, 443, (Synth. Chem. Agrochem. 2), 226-235. The reaction without the use of catalysts and solvents has been described by S. I. Hong et al. in J. Polym. Sci. Part A-1, 1972, 10, 3405-19. Even at very high temperatures, yields of only 33% are obtained. These processes have serious ecological and economic disadvantages, for example that a heterogeneous reaction mixture is obtained and lengthy reaction times of up to 10 hours are required. The use of a fluoride complicates the working up of the reaction mixture on an industrial scale. A filtration and a washing step must be carried out subsequently. In addition, the wash-water has to be reprocessed.

SUMMARY:

BSUM(5)

In one of its aspects, the invention relates to a process for the hydroxyalkylation of aldoximes and ketoximes by reacting said ****oximes**** with unsubstituted or C_{1-8} alkyl-substituted ethylene or ****propylene** carbonate**** in the presence of a catalyst, which comprises the use of catalytic amounts of a N-alkylated, stable, organic amidine base or a pyridine which is substituted by a secondary amino group.

DETDESC:

DETD(2)

A solution of 160 g (1.81 mol) of **ethylene carbonate** and 11.02 g (0.07 mol) of 1,8-diazabicyclo[5.4.0]undec-7-ene in 220 g of toluene is heated to reflux in a reactor fitted with reflux condenser, thermometer, dropping funnel and stirrer, and which is provided externally with a temper jacket. A solution of 146.2 g (2.00 mol) of acetone **oxime** in 217 g of toluene is added dropwise to this solution over a period of 2 hours. Afterwards the reaction solution is kept under reflux for 1 hour until the evolution of CO₂ gas has ceased and the conversion of **ethylene carbonate** is complete. Yield: 722 g of a 24.4 % solution of **oxime** glycol in toluene, corresponding to a yield of 83.1%. The pure **oxime** glycol is obtained in a yield of 65 % to 75 %, depending on the desired purity, by carrying out two subsequent rectifications.

DETDESC:

DETD(4)

The procedure of Example 1 is repeated, using 13.82 g (0.09 mol) of 1,8-diazabicyclo[5.4.0]undec-7-ene in 220 g of toluene and heating the mixture to reflux. A solution of 146.2 g (2.00 mol) of acetone **oxime** in 330 g of toluene is added dropwise to this solution over a period of 2 hours. The further steps of Example 1 are carried out, giving 813 g of a 21.4% solution of **oxime** glycol in toluene, corresponding to a yield of 82.1%. The pure **oxime** glycol is obtained in a yield of 65% to 75%, depending on the desired purity, by carrying out two subsequent rectifications.

CLAIMS:

CLMS(1)

What is claimed is:

1. A process for the hydroxyalkylation of aldoximes and ketoximes by reacting said **oximes** with unsubstituted or substituted ethylene or **propylene carbonate** in the presence of a catalyst, which comprises the use of catalytic amounts of a N-alkylated, stable, organic amidine base or a pyridine base which is substituted by a secondary amino group.

US PAT NO: 5,425,854 [IMAGE AVAILABLE]

L5: 8 of 29

ABSTRACT:

Methylene chloride is difficult to separate from tetrahydrofuran by conventional distillation or rectification because of the proximity of their vapor pressures. Methylene chloride can be readily separated from

tetrahydrofuran by extractive distillation. Effective agents are 1-pentanol, 1,2-butanediol and 3-nitrotoluene.

US PAT NO: 5,425,854 [IMAGE AVAILABLE] L5: 8 of 29
DATE ISSUED: Jun. 20, 1995
TITLE: Separation of methylene chloride from tetrahydrofuran by
extractive distillation
INVENTOR: Lloyd Berg, 1314 S. Third Ave., Bozeman, MT 59715
ASSIGNEE: Lloyd Berg, Bozeman, MT (U.S. indiv.)
APPL-NO: 08/373,549
DATE FILED: Jan. 17, 1995
ART-UNIT: 138
PRIM-EXMR: Wilbur Bascomb, Jr.

SUMMARY:

BSUM(16)

TABLE 3

Effective Extractive Distillation Agents For
Separating Methylene Chloride From Tetrahydrofuran

Compounds	Relative Volatility
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None	1.15
Ethylene glycol	1.65
1,2-Butanediol	2.2
1,3-Butanediol	2.2
1,4-Butanediol	1.7
2,3-Butanediol	2.0
Dipropylene glycol	1.6
1,5-Pentanediol	1.6
3-Methyl-2,4-pentanediol	1.9
2-Methyl-1,3-propanediol	1.75
1,2,6-Trihydroxyhexane	1.75
2-Hydroxyacetophenone	1.65
Benzyl benzoate	1.55
Butyl lactate	1.55
Phenyl acetate	1.65
Butyronitrile	1.5
Methyl ethyl ketoxime	

	2.0
Methyl isobutyl ketoxime	1.5
Ethyl caproate	1.5
Ethyl isovalerate	1.6
Butyl aldehyde **oxime**	2.5
Methyl valerate	1.7
Methyl benzoate	1.7
Benzonitrile	1.85
Isopropyl lactate	1.55
Isoamyl formate	1.65
Butyl formate	1.55
Butyl butyrate	1.55
Tri-2-ethyl hexyl trimellitate	1.5
Propylene **carbonate**	1.55
2-Undecanone	1.5
Diisobutyl ketone	1.55
2,6-Dimethyl-4-heptanone	1.6
Acetophenone	1.7
Nitromethane	1.7
Nitroethane	1.9
1-Nitropropane	1.9
2-Nitropropane	1.65
Nitrobenzene	1.75
2-Nitrotoluene	1.75
3-Nitrotoluene	2.2
Methyl n-amyl ketoxime	1.95
Acetonitrile	1.55
Butyronitrile	1.5
Tridecyl alcohol	1.5
1-Decanol	1.6
Isodecyl alcohol	1.95
2-Octanol	1.9
Phenethyl alcohol	1.85
1-Undecanol	1.75
1-Hexanol	2.2
Cyclopentanol	2.1
Cyclohexanol	1.85
1-Pentanol	2.25*

*Data obtained in multiplate rectification column

SUMMARY:

BSUM(17)

ethylene glycol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 2,3-butanediol, dipropylene glycol, 1,5-pentanediol, 3-methyl-2,4-pentanediol, 2-methyl-1,3-propanediol, 1,2,6-trihydroxyhexane, 2-hydroxyacetophenone, benzyl benzoate, butyl lactate, phenyl acetate, butyronitrile, methyl ethyl ketoxime, methyl isobutyl ketoxime, methyl n-amyl ketoxime, ethyl caproate, ethyl isovalerate, butyl aldehyde **oxime**, methyl valerate, methyl benzoate, benzonitrile, isopropyl lactate, isoamyl formate, butyl formate, butyl butyrate, tri-2-ethyl hexyl trimellitate, **propylene** **carbonate**, 2-undecanone, diisobutyl ketone, 2,6-dimethyl-4-heptanone, acetophenone, nitromethane, nitroethane, 1-nitropropane, 2-nitropropane, nitrobenzene, 2-nitrotoluene, 3-nitrotoluene, acetonitrile, butyronitrile, tridecyl alcohol, 1-decanol, isodecyl alcohol, 2-octanol, phenethyl alcohol, 1-undecanol, 1-hexanol, cyclopentanol, cyclohexanol and 1-pentanol.

CLAIMS:

CLMS(1)

I claim:

1. A method for recovering methylene chloride from a mixture of methylene chloride and tetrahydrofuran which comprises distilling a mixture of methylene chloride and tetrahydrofuran in the presence of about one part by weight of an extractive agent per part of methylene chloride-tetrahydrofuran mixture, recovering the methylene chloride as overhead product and obtaining the tetrahydrofuran and the extractive agent as bottoms product, wherein said extractive agent consists of one material selected from the group consisting of ethylene glycol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 2,3-butanediol, dipropylene glycol, 1,5-pentanediol, 3-methyl-2,4-pentanediol, 2-methyl-1,3-propanediol, 1,2,6-trihydroxyhexane, 2-hydroxyacetophenone, benzyl benzoate, butyl lactate, phenyl acetate, butyronitrile, methyl ethyl ketoxime, methyl isobutyl ketoxime, methyl n-amyl ketoxime, ethyl caproate, ethyl isovalerate, butyl aldehyde **oxime**, methyl valerate, methyl benzoate, benzonitrile, isopropyl lactate, isoamyl formate, butyl formate, butyl butyrate, tri-2-ethyl hexyl trimellitate, **propylene** **carbonate**, 2-undecanone, diisobutyl ketone, 2,6-dimethyl-4-heptanone, acetophenone, nitro-methane, nitroethane, 1-nitropropane, 2-nitropropane, nitrobenzene, 2-nitrotoluene, 3-nitrotoluene, acetonitrile, butyronitrile, tridecyl alcohol, 1-decanol, isodecyl alcohol, 2-octanol, phenethyl alcohol, 1-undecanol, 1-hexanol, cyclopentanol, cyclohexanol and 1-pentanol.

ABSTRACT:

A resin composition for investment casting contains a moderate boiling temperature liquid inert diluent, having a boiling point in the range of about 200.degree. to 300.degree. C. The presence of the liquid inert diluent allows the resin to be used as a pattern, or model, in investment casting by preventing breaking of the mold during burnout of the resin. Any of the conventional resins suitable for stereolithography may be employed in the practice of the invention. To the resin is added a moderate boiling point solvent in the amount of about 5 to 30 wt %. Use of the moderate boiling point solvent in conjunction with the resin allows the pattern to shrink and crack at moderate temperature before final burnout. The shrinkage occurs faster and at a lower temperature as compared to the prior art.

US PAT NO: 5,391,460 [IMAGE AVAILABLE] L5: 9 of 29
DATE ISSUED: Feb. 21, 1995
TITLE: Resin composition and process for investment casting using
stereolithography
INVENTOR: Thomas K. Dougherty, Playa Del Rey, CA
William E. Elias, El Segundo, CA
Timothy C. Thelander, Riverside, CA
Mahesh N. Bhavnani, Diamond Bar, CA
ASSIGNEE: Hughes Aircraft Company, Los Angeles, CA (U.S. corp.)
APPL-NO: 08/089,544
DATE FILED: Jul. 12, 1993
ART-UNIT: 157
PRIM-EXMR: Marion E. McCamish
ASST-EXMR: Mark A. Chapman
LEGAL-REP: M. E. Lachman, M. W. Sales, W. K. Denson-Low

SUMMARY:

BSUM(26)

Examples of photoactivated free radical photoinitiators employed include, but are not limited to, benzyl and phenyl ketones, benzyl and phenyl ketals benzoin ethers acetophenones, benzoyl ****oximes****, acylphosphines, xanthenes, and quinones, and the like, for example, benzophenone, .alpha..alpha.-dimethoxyphenylacetophenone, 1-hydroxycyclohexyl phenyl ketone, benzyldimethyl ketal, isopropylthioxanthone, and ethyl 4-(dimethylamino benzoate). Cationic photoinitiators used with vinyl ether resins include, but are not limited to, di- and tri-aryliodonium, sulfonium, and arsenic and selenium salts, such as triarylsulfonium hexafluoroantimonate, triarylsulfonium hexafluorophosphate, and 4,4'-di-t-butylldiphenyliodonium

hexafluoroarsenate.

SUMMARY:

BSUM(28)

In the practice of the present invention, a modified resin composition is formulated which adds to the above photopolymerizable mixture a moderate boiling point inert solvent in a concentration of between 5 and 30%. All amounts herein are in terms of weight percent, unless otherwise specified. Such a solvent is soluble in the resin composition, is inert to vinyls and radicals generated during the photochemical reaction, and has a boiling point ranging from about 200.degree. to 300.degree. C. A preferred solvent is ****propylene** carbonate****, which has a boiling point of 240.degree. C. Examples of other inert solvents suitably employed in the practice of the present invention include phenol, glycerol, ethylene glycol, diglyme, benzylethyl ether, phenylether, veratrole, ethylene glycol diacetate, benzyl acetate, benzyl alcohol, methylbenzoate, ethylbenzoate, butyrolactone, ethyl malonate, butyl phosphate, trichlorotrifluoroethane, chloronaphthalene, nitrobenzene, succinonitrile, tolunitrile, octanitrile, benzonitrile, aniline, toluidine, quiniline, and the like.

CLAIMS:

CLMS(2)

2. The stereolithographic resin of claim 1 wherein said moderate boiling point solvent is selected from the group consisting of ****propylene** carbonate****, phenol, glycerol, ethylene glycol, diglyme, benzylethyl ether, phenylether, veratrole, ethylene glycol diacetate, benzyl acetate, benzyl alcohol, methylbenzoate, ethylbenzoate, butyrolactone, ethyl malonate, butyl phosphate, trichlorotrifluoroethane, chloronaphthalene, nitrobenzene, succinonitrile, tolunitrile, octanitrile, benzonitrile, aniline, toluidine, and quinoline.

CLAIMS:

CLMS(4)

4. The stereolithographic resin of claim 1 wherein said photoinitiator comprises a photoactivated free radical initiator selected from the group consisting of benzyl and phenyl ketones, benzyl and phenyl ketals, benzoin ethers, acetophenones, benzoyl ****oximes****, acylphosphines, xanthenes, and quinones.

CLAIMS:

CLMS(11)

11. The method of claim 10 wherein said moderate boiling point solvent is selected from the group consisting of **propylene** **carbonate**, phenol, glycerol, ethylene glycol, diglyme, benzylethyl ether, phenylether, veratrole, ethylene glycol diacetate, benzyl acetate, benzyl alcohol, methylbenzoate, ethylbenzoate, butyrolactone, ethyl malonate, butyl phosphate, trichlorotrifluoroethane, chloronaphthalene, nitrobenzene, succinonitrile, tolunitrile, octanitrile, benzonitrile, aniline, toluidine, and quinoline.

CLAIMS:

CLMS(13)

13. The method of claim 10 wherein said photoinitiator comprises a photoactivated free radical initiator selected from the group consisting of benzyl and phenyl ketones, benzyl and phenyl ketals, benzoin ethers, acetophenones, benzoyl **oximes**, acylphosphines, xanthenes, and quinones.

CLAIMS:

CLMS(16)

16. The method of claim 15 wherein said inert solvent comprises **propylene** **carbonate**.

US PAT NO: 5,356,529 [IMAGE AVAILABLE]

L5: 10 of 29

ABSTRACT:

Electrodepositable compositions comprising active hydrogen-containing ionic resins, capped polyisocyanate curing agents and triorganotin catalysts are disclosed. The catalysts are liquid, can be easily incorporated into the electrodepositable compositions and surprisingly exhibit good catalytic activity at low levels.

US PAT NO: 5,356,529 [IMAGE AVAILABLE]

L5: 10 of 29

DATE ISSUED: Oct. 18, 1994

TITLE: Electrodepositable compositions containing triorganotin catalysts

INVENTOR: V. Eswarakrishnan, Allison Park, PA
Robert R. Zwack, Allison Park, PA
Edward R. Coleridge, Lower Burrell, PA

ASSIGNEE: PPG Industries, Inc., Pittsburgh, PA (U.S. corp.)

APPL-NO: 08/057,368
DATE FILED: May 3, 1993
ART-UNIT: 153
PRIM-EXMR: John Kight, III
ASST-EXMR: Jeffrey Culpeper Mullis
LEGAL-REP: William J. Uhl

SUMMARY:

BSUM(29)

The capping agent of the capped isocyanate groups can be an alcohol, including glycol monoethers and amino alcohols. Examples include aliphatic alcohols such as methanol and 2-ethylhexyl alcohol; cycloaliphatic alcohols such as cyclohexanol; aromatic alkyl alcohols such as benzyl alcohol; glycol monoethers such as the monoalkyl ethers of ethylene glycol, i.e. the monobutyl ether of diethylene glycol; and amino alcohols such as dimethylethanolamine. ****Oximes**** such as methyl ethyl ketoxime; lactams such as epsilon-caprolactam; aliphatic amines such as dibutylamine and beta-dicarbonyl compounds such as acetyl acetone can also be used.

SUMMARY:

BSUM(32)

Besides capped polyisocyanates which are prepared by reacting an organic polyisocyanate with a capping agent as described above, capped polyisocyanates which are formed by reacting a carbonate such as ethylene or ****propylene**** ****carbonate**** with a polyamine can also be used.

US PAT NO: 5,350,825 [IMAGE AVAILABLE]

L5: 11 of 29

ABSTRACT:

The present invention relates to organic polyisocyanates which have a content of unblocked and blocked isocyanate groups (calculated as NCO) of 5 to 20 wt.%, preferably 7 to 15 wt.%, wherein at least 95%, preferably 100%, of the isocyanate groups are present in blocked form and wherein the blocking agent contains

- A) 30 to 70 equivalent-% of diisopropylamine,
- B) 30 to 70 equivalent-% of at least one other blocking agent selected from CH-acidic esters and 1,2,4-triazole and
- C) 0 to 20 equivalent-% of blocking agents other than those set forth in A) and B),

wherein the percentages of A), B) and C) add up to 100.

The present invention also relates to a process for the production of these organic polyisocyanates and their use as crosslinking agents for

organic polyhydroxyl compounds in polyurethane coating compositions.

US PAT NO: 5,350,825 [IMAGE AVAILABLE] L5: 11 of 29
DATE ISSUED: Sep. 27, 1994
TITLE: At least partially blocked organic polyisocyanates, a
process for their preparation and their use in coating
compositions
INVENTOR: Eberhard Konig, Cologne, Federal Republic of Germany
Holger Casselmann, Bergisch Gladbach, Federal Republic of
Germany
Frank Kobelka, Leverkusen, Federal Republic of Germany
Keith-Allen Foster, Krefeld, Federal Republic of Germany
ASSIGNEE: Bayer Aktiengesellschaft, Leverkusen, Federal Republic of
Germany (foreign corp.)
Miles Inc., Pittsburgh, PA (U.S. corp.)
APPL-NO: 08/157,350
DATE FILED: Nov. 23, 1993
ART-UNIT: 153
PRIM-EXMR: Maurice J. Welsh
LEGAL-REP: Joseph C. Gil, Thomas W. Roy, Richard E. L. Henderson

DETDESC:

DETD(5)

The starting polyisocyanates may also be in prepolymer form by reacting the preceding polyisocyanates with polyhydric alcohols having an OH number of from 56 to 1900. To prepare the prepolymers 0,05 to 0,6 equivalents of OH compounds are reacted per equivalent of NCO groups. Examples of such polyols are 1,4dihydroxybutane, 2-ethyl-1,3-hexanediol, tri- and tetrapropylene glycol, other polypropylene glycols or polypropylene glycol mixtures having the necessary OH number and the addition product of 2 moles of **propylene** **carbonate** onto 1 mole of hydrafine-Trifunctional polyols such as glycerol or trimethylol propane may also be used, optionally in admixture with the diols exemplified hereinbefore. Among the preferred prepolymers are those which are based on 2,4-diisocyanato toluene and a polyol mixture comprising (i) polypropylene glycol having an OH number of from 56 to 112, (ii) from 50 to 150 OH-equivalent-%, based on (i), of diethylene glycol and (iii) from 150 to 250 OH-equivalent-% based on (i), of trimethylol propane and which are prepared at an NCO/OH-equivalent ratio of from 1,6:1 to 2:1. Prepolymer formation of the starting polyisocyanates may proceed either simultaneously with the blocking reaction, for example, by reacting the starting polyisocyanates with a mixture of blocking agents and polyol, or sequentially by reacting the starting polyisocyanate with the blocking agents and the polyol in any desired order. The blocking agents to be

used in the process according to the invention contain, based on the total equivalents of blocking agent, 30 to 70, preferably 40 to 60, equivalent-% of blocking agent A), 30 to 70, preferably 40 to 60 equivalent-%, of blocking agent B) and 0 to 20, preferably 0, equivalent-% of blocking agent C).

DETDESC:

DETD(7)

Other suitable blocking agents which may be used in conjunction with blocking agents A) and B) are known and include butanone **oxime** and .epsilon.-caprolactam.

US PAT NO: 5,232,988 [IMAGE AVAILABLE]

L5: 12 of 29

ABSTRACT:

The present invention relates to a blocked polyisocyanate which is based on the reaction product of a polyisocyanate with a reversible, monofunctional blocking agent for isocyanate groups, wherein the polyisocyanate is prepared by trimerizing 5 to 85% of the isocyanate groups of a cyclic organic diisocyanate having (cyclo)aliphatically bound isocyanate groups and contains

- i) an isocyanurate group-containing polyisocyanate and
- ii) at least 5% by weight, based on the weight of the polyisocyanate, of unreacted diisocyanate.

The present invention also relates to a one-component coating composition containing this blocked polyisocyanate and a polyhydroxyl polyacrylate and/or a polyhydroxyl polyester.

Finally, the present invention relates to substrates coated with this coating composition.

US PAT NO: 5,232,988 [IMAGE AVAILABLE]

L5: 12 of 29

DATE ISSUED: Aug. 3, 1993

TITLE: Blocked polyisocyanates prepared from partially trimerized cyclic organic diisocyanates having (cyclo)aliphatically bound isocyanate groups and their use for the production of coatings

INVENTOR: Lanny D. Venham, Paden City, WV
Marianne M. Salek, Oakdale, PA
Terry A. Potter, Beaver, PA

ASSIGNEE: Miles Inc., Pittsburgh, PA (U.S. corp.)

APPL-NO: 07/841,130

DATE FILED: Feb. 25, 1992

ART-UNIT: 153

PRIM-EXMR: Maurice J. Welsh

LEGAL-REP: Joseph C. Gil, Thomas W. Roy

DETDESC:

DETD(21)

Preferred blocking agents are the ****oximes****; methyl ethyl ketoxime is especially preferred.

DETDESC:

DETD(24)

Polycarbonates containing hydroxyl groups include those known such as the products obtained from the reaction of diols such as propanediol-(1,3), butanediol-(1,4) and/or hexanediol-(1,6), diethylene glycol, triethylene glycol or tetraethylene glycol with phosgene, diarylcarbonates such as diphenylcarbonate or with cyclic carbonates such as ethylene or ****propylene** carbonate****. Also suitable are polyester carbonates obtained from the above-mentioned polyesters or polylactones with phosgene, diaryl carbonates or cyclic carbonates.

US PAT NO: 5,147,490 [IMAGE AVAILABLE]

L5: 13 of 29

ABSTRACT:

A process for the production of a heat-mode recording material by coating in a sealed zone maintained under vacuum conditions a web support with a heat-mode recording layer of vapor depositable material and within the sealed zone laminating a protective organic resin layer in web form by means of an adhesive layer onto the supported heat-mode recording layer. The web support moves within the sealed zone from a delivery reel to a take-up roll with the recording layer deposited thereon in a region between the rolls and the protective layer is laminated over the recording layer before the coated web support is collected on the take-up rolls.

US PAT NO: 5,147,490 [IMAGE AVAILABLE]

L5: 13 of 29

DATE ISSUED: Sep. 15, 1992

TITLE: Process for the production of a heat-mode recording material

INVENTOR: Luc H. Leenders, Herentals, Belgium
Luciaan F. Voet, St. Katelijne-Waver, Belgium
Dirk M. D'hont, Mortsel, Belgium

ASSIGNEE: Agfa-Gevaert N.V., Mortsel, Belgium (foreign corp.)

APPL-NO: 07/480,603

DATE FILED: Feb. 15, 1990
ART-UNIT: 134
PRIM-EXMR: David A. Simmons
ASST-EXMR: Chester T. Barry
LEGAL-REP: William J. Daniel

DETDESC:

DETD(27)

According to a further embodiment the recording layer is present on an underlying layer containing a blowing agent and/or such blowing agent is present in the pressure-sensitive adhesive layer of the laminate produced according to the present invention. A blowing agent is a substance that decomposes and yields gaseous products by heating above 50.degree. C. Such substances are e.g. carbonates such as ammonium hydrogen carbonate, sodium carbonate and **ethylene** **carbonate** and sulphonylhydrazides. Further are mentioned urea, **oximes** such as 1,3,5-cyclohexanetrione trioxime and the photosensitive 0-acyl **oximes** described in U.S. Pat. No. 3,558,309.

US PAT NO: 5,112,877 [IMAGE AVAILABLE]

L5: 14 of 29

ABSTRACT:

Polyisocyanate composition comprising an organo clay and a cyclic alkylene carbonate. The composition is useful for wood-binding, preparing polyurethanes and rigid and flexible polyurethane foams.

US PAT NO: 5,112,877 [IMAGE AVAILABLE]

L5: 14 of 29

DATE ISSUED: May 12, 1992

TITLE: Polyisocyanate composition

INVENTOR: Martin C. Barker, Bertem, Belgium
James P. Brown, Sterrebeek, Belgium
Anthony Cunningham, Bertem, Belgium
David Randall, Erps-Kwerps, Belgium

ASSIGNEE: Imperial Chemical Industries PLC, London, England (foreign corp.)

APPL-NO: 07/409,703

DATE FILED: Sep. 20, 1989

ART-UNIT: 153

PRIM-EXMR: John Kight, III

LEGAL-REP: Cushman, Darby & Cushman

SUMMARY:

BSUM(9)

Cyclic alkylene carbonates are known as well. The alkylene chain of the polar activator used in the composition according to the present invention may have 3-10 and preferably 3-6 carbon atoms. Preferred compounds are butylene carbonate and **propylene carbonate**. Most preference is given to **propylene carbonate**.

SUMMARY:

BSUM(11)

Examples of organic polyisocyanates which may be used in the process of the invention include aliphatic isocyanates such as hexamethylene diisocyanate, aromatic isocyanates such as meta- and para-phenylene diisocyanate, tolylene-2,4- and 2,6-diisocyanates, diphenylmethane-4,4'-diisocyanate, chlorophenylene-2,4-diisocyanate, naphthylene-1,5-diisocyanate, diphenylene-4,4'-diisocyanate, 4,4'-diisocyanate-3,3'-dimethyldiphenyl, 3-methyldiphenylmethane-4,4'-diisocyanate and diphenyl ether diisocyanate, cycloaliphatic diisocyanates such as cyclohexane-2,4- and 2,3-diisocyanates, 1-methyl cyclohexyl-2,4- and 2,6-diisocyanates and mixtures thereof and bis- (isocyanatocyclohexyl)-methane and tri- isocyanates such as 2,4,6-triisocyanatotoluene and 2,4,4-triisocyanatodiphenylether. Modified polyisocyanates, like those containing isocyanurate-, carbodiimide-, uretonimine-, urea-, biuret-, allophanate- or uretidinedione groups may be employed as well. Further blocked polyisocyanates, like the reaction product of a phenol or an **oxime** and a polyisocyanate, having a deblocking temperature below the temperature applied using the composition according to the present invention, may be used.

US PAT NO: 5,039,720 [IMAGE AVAILABLE]

L5: 15 of 29

ABSTRACT:

An aqueous electrophoretic coating material, which can be deposited at the cathode and contains

- (A) 90 to 50% by weight, based on the total portion of synthetic resin vehicle, of one or several polycondensation, polymerization and/or poly-addition resins, the resin or, in the event that several resins are present, their mixture
 - (a) having a number average molecular weight (Mn) of 700 to 10,000
 - (b) having 1.2.times.10.sup.23 to 21.7.times.10.sup.23 lateral or terminal double bonds per 1,000 g, corresponding to a hydrogenation iodine number of 5 to 90,
 - (c) optionally containing primary and/or secondary hydroxyl groups, corresponding to a hydroxyl number of 0 to 450 mg KOH/g of resin, and
 - (d) tertiary amino groups corresponding to an amine number of 30 to 450 mg KOH/g of resin.

(B) 10 to 50% by weight of one or several cross linking agents, which are compounds with methane tricarboxylic acid amide units of the formula ##STR1## which contain, on the average, at least 2 groups with reactive hydrogens per molecule and are derived from a reaction product of a monoisocyanate or polyisocyanate with a malonic acid derivative, and

(C) 0 to 10% by weight of one or several capped diisocyanates and/or polyisocyanates, as well as its use.

US PAT NO: 5,039,720 [IMAGE AVAILABLE] L5: 15 of 29
DATE ISSUED: Aug. 13, 1991
TITLE: Aqueous electrophoretic enamel coating materials, which
can be deposited at the cathode crosslinked with methane
tricarboxylic acid amides of malonic acid derivatives
INVENTOR: Dietrich Saatweber, Wuppertal, Federal Republic of Germany
Georg Hendriux, Wuppertal, Federal Republic of Germany
Gerhard Brindopke, Sulzbach/Ts, Federal Republic of
Germany
Helmut Plum, Taunusstein, Federal Republic of Germany
ASSIGNEE: Hoechst Aktiengesellschaft, Frankfurt, Federal Republic of
Germany (foreign corp.)
APPL-NO: 07/251,673
DATE FILED: Sep. 30, 1988
ART-UNIT: 153
PRIM-EXMR: Earl Nielsen
ASST-EXMR: Frederick Krass
LEGAL-REP: Schweitzer Cornman & Gross

DETDESC:

DETD(47)

As blocking or capping agents, alcohols, phenols, thioalcohols, thiophenols, **oximes**, hydroxamate esters, hydroxyalkyl (meth)acrylates, alkanolamines such as dimethylethanolamine, diethanolamine, etc., amines, amides, imides and/or .beta.-dicarbonyl compounds may be used.

DETDESC:

DETD(48)

A further possibility for synthesizing capped or blocked isocyanates lies in the reaction of diamines or polyamines with alkylene carbonates, as a result of which .beta.-hydroxyurethanes are formed. These reactions are described in the DE-A-32 46 812, page 3, line 29 to page 4, line 8.

According to the DE-A-32 46 812, the polyamines are reacted only partially with alkylene carbonate. It is, however, obvious to those skilled in the art that, for example, all 3 amino groups of the diethylenetriamine molecule can be reacted with alkylene carbonate, for example with ****propylene** carbonate****. Especially preferred for the present invention are capped prepolymers of aliphatic triol, aliphatic-aromatic diisocyanate and aliphatic ketoxime.

US PAT NO: 5,015,673 [IMAGE AVAILABLE]

L5: 16 of 29

ABSTRACT:

A synthetic resin which carries basic nitrogen groups and is water-dilutable by protonation with acid is obtainable by reacting

- (A) an epoxy resin having an average molecular weight $M_{sub.n}$ of from 300 to 6,000 and on average from 1.5 to 3.0 epoxy groups per molecule with
- (B) a ketimine of a primary amine additionally containing one or more urethane groups and optionally also with
- (C) a diketimine of a diprimary amine containing no urethane groups and optionally also with
- (D) a secondary amine which may contain a tertiary amino group also, a ketimine of a primary monoamine and/or a ketimine of a primary/tertiary diamine.

US PAT NO: 5,015,673 [IMAGE AVAILABLE]

L5: 16 of 29

DATE ISSUED: May 14, 1991

TITLE: Synthetic resin with basic nitrogen groups, preparation and use thereof using a urethane carrying ketimine

INVENTOR: Thomas Perner, Ludwigshafen, Federal Republic of Germany
Rolf Osterloh, Erftstadt, Federal Republic of Germany
Eberhard Schupp, Gruenstadt, Federal Republic of Germany
Thomas Schwerzel, Ludwigshafen, Federal Republic of Germany

Klaas Ahlers, Muenster, Federal Republic of Germany

ASSIGNEE: BASF Lacke & Farben Aktiengesellschaft, Muenster, Federal Republic of Germany (foreign corp.)

APPL-NO: 07/528,374

DATE FILED: May 25, 1990

ART-UNIT: 153

PRIM-EXMR: Earl Nielsen

ASST-EXMR: Frederick Krass

DETDESC:

DETD(27)

Suitable crosslinking agents for the binder according to the invention

are for example aminoplast resins, such as urea-formaldehyde resins, melamine resins or benzoguanamine resins, phenoplast resins, blocked isocyanate crosslinking agents, crosslinking agents which act via esteraminolysis and/or transesterification and have an average of at least two activated ester groups per molecule, for example .beta.-hydroxyalkyl ester crosslinking agents as described in EP 0,040,867 and carboalkoxymethyl ester crosslinking agents as described in German Laid-Open Application No. DOS 3,233,139, urea condensation products, as obtained for example from primary diamines and/or polyamines, secondary monoamines and urea, in the presence or absence of a polyalcohol, for example trimethylolpropane, and .beta.-hydroxyalkyl carbamate crosslinking agents as obtained for example by reacting polyprimary amines with **ethylene** **carbonate**. Particular preference is given to crosslinking agents which contain urethane groups, for example alcohol-capped isocyanates which are particularly readily dispersible in combination with the claimed binders and show very good stability not only in unpigmented but also in pigmented form.

DETDESC:

DETD(29)

It is not absolutely necessary to add separate crosslinking agents since crosslinking agent functions can also be incorporated in the synthetic resin according to the invention, so that it becomes self-crosslinking. This can be effected for example after the reaction of (A), (B) and if used (C) and (D) by reacting the resulting resin with a partially blocked polyisocyanate which on average still contains about one free isocyanate group in the molecule. The capping agents used on the polyisocyanate can be for example alcohols, phenols and **oximes**. A further way of rendering the synthetic resin according to the invention self-crosslinking consists in introducing .beta.-hydroxyalkyl carbamate groups. To this end the ketone is first eliminated by hydrolysis from the resin according to the invention. The resulting primary amino groups are then for example converted with **ethylene** **carbonate** into .beta.-hydroxyethyl carbamates.

US PAT NO: 5,015,672 [IMAGE AVAILABLE]

L5: 17 of 29

ABSTRACT:

A synthetic resin which carries basic nitrogen groups and is water-dilutable by protonation with acid is obtainable by reacting (A) an epoxy resin having an average molecular weight $M_{sub.n}$ of from 300 to 6,000 and on average from 1.5 to 3.0 epoxy groups per molecule with (B) a ketimine of a primary amine additionally containing one or more urethane groups and optionally also with (C) a diketimine of a diprimary amine containing no urethane groups and

optionally also with

(D) a secondary amine which may contain a tertiary amino group also, a ketimine of a primary monoamine and/or a ketimine of a primary/tertiary diamine.

US PAT NO: 5,015,672 [IMAGE AVAILABLE] L5: 17 of 29
DATE ISSUED: May 14, 1991
TITLE: Synthetic resin with basic nitrogen groups, preparation
and use thereof using a urethane carrying ketimine
INVENTOR: Thomas Perner, Ludwigshafen, Federal Republic of Germany
Rolf Osterloh, Erftstadt, Federal Republic of Germany
Eberhard Schupp, Gruenstadt, Federal Republic of Germany
Thomas Schwerzel, Ludwigshafen, Federal Republic of
Germany
Klaas Ahlers, Muenster, Federal Republic of Germany
ASSIGNEE: BASF Lacke & Farben Aktiengesellschaft, Muenster, Federal
Republic of Germany (foreign corp.)
APPL-NO: 07/528,377
DATE FILED: May 25, 1990
ART-UNIT: 153
PRIM-EXMR: Earl Nielsen
ASST-EXMR: Frederick Krass
LEGAL-REP: Keil & Weinkauff

SUMMARY:

BSUM(44)

Suitable crosslinking agents for the binder according to the invention are for example aminoplast resins, such as urea-formaldehyde resins, melamine resins or benzoguanamine resins, phenoplast resins, blocked isocyanate crosslinking agents, crosslinking agents which act via esteraminolysis and/or transesterification and have an average of at least two activated ester groups per molecule, for example .beta.-hydroxyalkyl ester crosslinking agents as described in EP No. 0,040,867 and carboalkoxymethyl ester crosslinking agents as described in German Laid-Open Application DOS No. 3,233,139, urea condensation products, as obtained for example from primary diamines and/or polyamines, secondary monoamines and urea, in the presence or absence of a polyalcohol, for example trimethylolpropane, and .beta.-hydroxyalkyl carbamate crosslinking agents as obtained for example by reacting polyprimary amines with **ethylene** **carbonate**. Particular preference is given to crosslinking agents which contain urethane groups, for example alcohol-capped isocyanates which are particularly readily dispersible in combination with the claimed binders and show very good stability not only in unpigmented but also in pigmented form.

SUMMARY:

BSUM(46)

It is not absolutely necessary to add separate crosslinking agents since crosslinking agent functions can also be incorporated in the synthetic resin according to the invention, so that it becomes self-crosslinking. This can be effected for example after the reaction of (A), (B) and if used (C) and (D) by reacting the resulting resin with a partially blocked polyisocyanate which on average still contains about one free isocyanate group in the molecule. The capping agents used on the polyisocyanate can be for example alcohols, phenols and ****oximes****. A further way of rendering the synthetic resin according to the invention self-crosslinking consists in introducing .beta.-hydroxyalkyl carbamate groups. To this end the ketone is first eliminated by hydrolysis from the resin according to the invention. The resulting primary amino groups are then for example converted with ****ethylene** carbonate**** into .beta.-hydroxyethyl carbamates.

US PAT NO: 4,981,884 [IMAGE AVAILABLE]

L5: 18 of 29

ABSTRACT:

A synthetic resin which carries basic nitrogen groups and is water-dilutable by protonation with an acid, containing

(A) a chain-extended, amine-modified epoxy resin obtainable by reacting
(a.sub.1) an epoxy resin having an average molecular weight M.sub.n of from 300 to 6,000 and on average from 1.5 to 3.0 epoxy groups per molecule with

(a.sub.2) a diketimine of a diprimary diamine, which diprimary diamine contains no further groups reactive with epoxy groups under mild conditions and

(B) an amino-modified epoxy resin obtainable by reacting

b.sub.1) an epoxy resin having an average molecular weight M.sub.n of from 800 to 6,000 and on average from 1.5 to 3.0 epoxy groups per molecule with

(b.sub.2) a secondary amine or a secondary-tertiary diamine, a ketimine of a primary monoamine or a ketimine of a primary/tertiary diamine or a mixture of these (b.sub.2) components, with the proviso that (A) and (B) are different.

US PAT NO: 4,981,884 [IMAGE AVAILABLE]

L5: 18 of 29

DATE ISSUED: Jan. 1, 1991

TITLE: Synthetic resin with basic nitrogen groups, preparation and use thereof

INVENTOR: Thomas Perner, Ludwigshafen, Federal Republic of Germany
Rolf Osterloh, Erftstadt, Federal Republic of Germany
Eberhard Schupp, Gruenstadt, Federal Republic of Germany
Thomas Schwerzel, Ludwigshafen, Federal Republic of
Germany
Klaas Ahlers, Muenster, Federal Republic of Germany
ASSIGNEE: BASF Aktiengesellschaft, Ludwigshafen, Federal Republic of
Germany (foreign corp.)
APPL-NO: 07/210,502
DATE FILED: Jun. 23, 1988
ART-UNIT: 151
PRIM-EXMR: Lewis T. Jacobs
ASST-EXMR: Karen A. Hellender
LEGAL-REP: Keil & Weinkauff

SUMMARY:

BSUM(48)

Suitable crosslinking agents for the binder according to the invention are for example aminoplast resins, such as urea-formaldehyde resins, melamine resins or benzoguanamine resins, phenoplast resins, blocked isocyanate crosslinking agents, crosslinking agents which act via esteraminolysis and/or transesterification and have an average of at least two activated ester groups per molecule, for example .beta.-hydroxyalkyl ester crosslinking agents as described in EP No. 0,040,867 and carbalkoxymethyl ester crosslinking agents, urea condensation products, as obtained for example from primary diamines and/or polyamines, secondary monoamines and urea, in the presence or absence of a polyalcohol, for example trimethylolpropane, and .beta.-hydroxyalkyl carbamate crosslinking agents as obtained for example by reacting polyprimary amines with **ethylene** **carbonate**. The amount of crosslinking agent to be used depends on the nature and number of groups to be reacted with one another in the synthetic resin and the crosslinking agent and on the crosslinking density desired. In general, synthetic resin:crosslinking agent ratios of from 1:9 to 9:1, preferably from 1:1 to 9:1, in particular from 1.5:1 to 4:1, each based on the weight, are used.

SUMMARY:

BSUM(49)

It is not absolutely necessary to add separate crosslinking agents since crosslinking agent functions can also be incorporated in the synthetic resin according to the invention, so that it becomes self-crosslinking. This can be effected for example by reacting the resulting resin (A) and

(B) with a partially blocked polyisocyanate which on average still contains about one free isocyanate group in the molecule. The capping agents used on the polyisocyanate can be for example alcohols, phenols and ****oximes****. A further way of rendering the synthetic resin according to the invention self-crosslinking consists in introducing .beta.-hydroxyalkyl carbamate groups. To this end the ketone is first eliminated by hydrolysis from the resin according to the invention. The resulting primary amino groups are then for example converted with ****ethylene** carbonate**** into .beta.-hydroxyethyl carbamates.

US PAT NO: 4,956,402 [IMAGE AVAILABLE]

L5: 19 of 29

ABSTRACT:

A synthetic resin which carries basic nitrogen groups and is water-dilutable by protonation with acid is obtainable by reacting

(A) an epoxy resin having an average molecular weight $M_{\text{sub}.n}$ of from 200 to 6,000 and on average from 1.5 to 3.0 epoxy groups per molecule with

(B) a ketimine of a primary amine additionally containing one or more urethane groups and optionally also with

(C) a diketimine of a diprimary amine containing no urethane groups and optionally also with

(D) a secondary amine which may contain a tertiary amino group also, a ketimine of a primary monoamine and/or a ketimine of a primary/tertiary diamine.

US PAT NO: 4,956,402 [IMAGE AVAILABLE]

L5: 19 of 29

DATE ISSUED: Sep. 11, 1990

TITLE: Synthetic resin with basic nitrogen groups from resins reacted with the ketimine-urethane product of an alkanolamine, a ketone and anisocyanate

INVENTOR: Thomas Perner, Ludwigshafen, Federal Republic of Germany
Rolf Osterloh, Erftstadt, Federal Republic of Germany
Eberhard Schupp, Gruenstadt, Federal Republic of Germany
Thomas Schwerzel, Ludwigshafen, Federal Republic of Germany

ASSIGNEE: Klaas Ahlers, Muenster, Federal Republic of Germany
BASF Lacke & Farben Aktiengesellschaft, Muenster, Federal Republic of Germany (foreign corp.)

APPL-NO: 07/210,499

DATE FILED: Jun. 23, 1988

ART-UNIT: 153

PRIM-EXMR: John Kight, III

ASST-EXMR: Frederick Krass

LEGAL-REP: Keil & Weinkauff

SUMMARY:

BSUM(44)

Suitable crosslinking agents for the binder according to the invention are for example aminoplast resins, such as urea-formaldehyde resins, melamine resins or benzoguanamine resins, phenoplast resins, blocked isocyanate crosslinking agents, crosslinking agents which act via esteraminolysis and/or transesterification and have an average of at least two activated ester groups per molecule, for example .beta.-hydroxyalkyl ester crosslinking agents as described in EP 0,040,867 and carboalkoxymethyl ester crosslinking agents as described in German Laid-Open Application DOS 3,233,139, urea condensation products, as obtained for example from primary diamines and/or polyamines, secondary monoamines and urea, in the presence or absence of a polyalcohol, for example trimethylolpropane, and .beta.-hydroxyalkyl carbamate crosslinking agents as obtained for example by reacting polyprimary amines with **ethylene** **carbonate**. Particular preference is given to crosslinking agents which contain urethane groups, for example alcohol-capped isocyanates which are particularly readily dispersible in combination with the claimed binders and show very good stability not only in unpigmented but also in pigmented form.

SUMMARY:

BSUM(46)

It is not absolutely necessary to add separate crosslinking agents since crosslinking agent functions can also be incorporated in the synthetic resin according to the invention, so that it becomes self-crosslinking. This can be effected for example after the reaction of (A), (B) and if used (C) and (D) by reacting the resulting resin with a partially blocked polyisocyanate which on average still contains about one free isocyanate group in the molecule. The capping agents used on the polyisocyanate can be for example alcohols, phenols and **oximes**. A further way of rendering the synthetic resin according to the invention self-crosslinking consists in introducing .beta.-hydroxyalkyl carbamate groups. To this end the ketone is first eliminated by hydrolysis from the resin according to the invention. The resulting primary amino groups are then for example converted with **ethylene** **carbonate** into .beta.-hydroxyethyl carbamates.

US PAT NO: 4,921,839 [IMAGE AVAILABLE]

L5: 20 of 29

ABSTRACT:

Antibacterially active 11,12-carbonate derivatives of erythromycin

9-(optionally substituted)oxime and 9-imino compounds and their pharmaceutically acceptable ester or acid addition salt thereof: ##STR1## wherein R.sup.1 denotes an oxime group, a substituted oxime group, or an imino group; R.sup.3 denotes a hydrogen atom or an unsubstituted or substituted alkyl group; R.sup.7 denotes hydrogen or methyl; one of R.sup.8 and R.sup.9 denotes hydrogen, hydroxy, alkoxy, alkanoyloxy, amino, substituted amino, or a group of the formula R.sup.A --SO.sub.2 --O--, in which R.sup.A denotes an organic group, and the other of R.sup.8 and R.sup.9 denotes hydrogen, or R.sup.8 and R.sup.9 together denote an oxo group, an oxime group, or a substituted oxime group.

US PAT NO: 4,921,839 [IMAGE AVAILABLE] L5: 20 of 29
DATE ISSUED: May 1, 1990
TITLE: Erythromycin a 11,12-carbonate 9-oxime derivatives
INVENTOR: Edward G. Brain, Betchworth, England
Eric Hunt, Betchworth, England
Andrew K. Forrest, Betchworth, England
ASSIGNEE: Beecham Group p.l.c., England (foreign corp.)
APPL-NO: 07/158,543
DATE FILED: Feb. 22, 1988
ART-UNIT: 183
PRIM-EXMR: Johnnie R. Brown
ASST-EXMR: Elli Peselev
LEGAL-REP: Jacobs & Jacobs

SUMMARY:

BSUM(16)

All of the above 11,12-carbonate derivatives are prepared by the reaction of an appropriate erythromycin compound with **ethylene** **carbonate** in the presence of a base, and a modified method of carrying out that process has recently been described (EP 0 119 431, Polfa).

SUMMARY:

BSUM(18)

Despite the many references to 11,12-carbonates of erythromycin and its derivatives, there has been no disclosure of 11,12-carbonates of erythromycin 9-**oxime** and 9-**oxime** ethers. Our attempts to prepare erythromycin A 9-**oxime** 11,12-carbonate both by reaction of

****ethylene** **carbonate**** with erythromycin 9-****oxime**** analogously to the above method and by conversion of the 9-keto group of erythromycin A 11,12-carbonate have met with failure.

SUMMARY:

BSUM(19)

We have now found that it is possible to prepare 11,12-carbonate derivatives of erythromycin 9-****oxime**** and 9-****oxime**** ethers by another method.

SUMMARY:

BSUM(20)

The present invention provides antibacterially active 11,12-carbonate derivatives of erythromycin 9-(optionally substituted)****oxime**** and 9-imino compounds.

SUMMARY:

BSUM(21)

In particular, the present invention provides a compound of the general formula I or a pharmaceutically acceptable ester or acid addition salt thereof: ##STR3## wherein R.sup.1 denotes an ****oxime**** group, a substituted ****oxime**** group, or an imino group;

US PAT NO: 4,710,561 [IMAGE AVAILABLE]

L5: 21 of 29

ABSTRACT:

A resin coating composition comprising an epoxy resin derivative having an imidazoline ring functional group of the formula ##STR1## wherein R.sub.1 represents an alkylene group having 2 to 6 carbon atoms, each of R.sub.2, R.sub.3, R.sub.4 and R.sub.5 represents a hydrogen atom or a methyl group, and R.sub.6 represents a residue of a carboxylic acid, as a resin binder. This resin coating composition is useful for cationic electrodeposition coating.

US PAT NO: 4,710,561 [IMAGE AVAILABLE]

L5: 21 of 29

DATE ISSUED: Dec. 1, 1987

TITLE: [Poly]imidazoline adduct for electrocoating

INVENTOR: Akira Tominaga, Hiratsuka, Japan
Reizihiro Nishida, Hiratsuka, Japan

ASSIGNEE: Kansai Paint Co., Ltd., Hyogo, Japan (foreign corp.)

APPL-NO: 06/935,306
DATE FILED: Nov. 26, 1986
ART-UNIT: 153
PRIM-EXMR: John Kight
ASST-EXMR: Frederick Krass
LEGAL-REP: Wenderoth, Lind & Ponack

DETDESC:

DETD(7)

475 Parts (0.5 mole) of bisphenol A-type epoxy resin having an epoxy equivalent of 475 (Epikote 1001, a tradename for a product of Yuka-Shell Co., Ltd.) was dissolved under heat in 235 parts of ethylene glycol monobutyl ether, and 47.5 parts (0.5 mole) of an 80% aqueous solution of hydroxyethylhydrazine was added. The mixture was reacted at 70.degree. C. until the amine value of the product decreased to not more than 1. ****Ethylene** **carbonate**** (44 parts; 0.5 mole) was added, and the mixture was reacted at 90.degree. C. until the amine value of the product no longer decreased. Then, 219 parts (0.5 mole) of dimeric acid/bis-N-hydroxyethylimidazoline reaction product having an amine value of 154 (Hartall M-34, a tradename for a product of Harima Chemical Co., Ltd.) was added to the reaction mixture, and the mixture was reacted at 95.degree. C. until there was no rise in viscosity. As a result, an epoxy resin derivative in accordance with the invention was obtained.

DETDESC:

DETD(8)

The epoxy resin derivative (118 parts; resin solids 90 parts), 10 parts of methy ethyl ketone ****oxime**** blocked isophorone diisocyanate and 1 part of polypropylene glycol (PP-4000) were mixed, and 1.28 parts (neutralization value 12) of acetic acid was added. The mixture was heated to about 60.degree. C., and with stirring, deionized water was gradually added to disperse the epoxy resin derivative in water to give an emulsion having a resin solids content of 30%, an average particle diameter of 0.2 micron and a pH of 5.8. The emulsion had good stability.

US PAT NO: 4,699,937 [IMAGE AVAILABLE]

L5: 22 of 29

ABSTRACT:

A synthetic resin carrying basic nitrogen groups, its preparation, coating materials prepared from this resin, and a process for cathodic electrocoating.

This synthetic resin is obtained by reacting

(A) an epoxy resin which

(B) a diketimine of a diprimary amine

and, if required,

(C) with a secondary amine, which may additionally contain a tertiary amino group, with a ketimine of a primary monoamine and/or with a ketimine of a primary/tertiary diamine,

a diketimine obtained from

(B.sub.1) a ketone and

(B.sub.2) a branched aliphatic diprimary diamine of 5 to 9 carbon atoms and/or

(B.sub.3) one or more diaminodiamides which are obtained by condensation of 2 moles of diamine (B.sub.2) and 1 mole of a dicarboxylic acid being employed as component (B).

After protonation with an acid, this synthetic resin is useful as a binder for cathodic electrocoating.

US PAT NO: 4,699,937 [IMAGE AVAILABLE]

L5: 22 of 29

DATE ISSUED: Oct. 13, 1987

TITLE: Synthetic resin carrying basic nitrogen groups, its preparation, coating materials produced using this resin, and cathodic electrocoating

INVENTOR: Werner Loch, Erpolzheim, Federal Republic of Germany
Eberhard Schupp, Schwetzingen, Federal Republic of Germany
Rolf Osterloh, Gruenstadt, Federal Republic of Germany
Klaas Ahlers, Muenster, Federal Republic of Germany

ASSIGNEE: BASF Farben+Fasern Aktiengesellschaft, Hamburg, Federal Republic of Germany (foreign corp.)

APPL-NO: 06/808,281

DATE FILED: Dec. 12, 1985

ART-UNIT: 155

PRIM-EXMR: Paul R. Michl

LEGAL-REP: Keil & Weinkauff

SUMMARY:

BSUM(50)

Examples of suitable crosslinking agents for the novel binder are aminoplast resins, such as urea/formaldehyde resins, melamine resins or benzoguanamine resins, phenoplast resins, blocked isocyanate crosslinking agents, crosslinking agents which are curable via ester aminolysis and/or transesterification and possess on average two or more active ester groups per molecule, e.g. .beta.-hydroxyalkyl ester crosslinking agents according to European Pat. No. 40,867, and carbalkoxymethyl ester crosslinking agents according to German Patent Application No. P 32 33 139.8, urea condensates, as obtained, for example, from primary di- and/or polyamines, secondary monoamines and urea, in the presence or

absence of a polyalcohol, e.g. trimethylolpropane, and .beta.-hydroxyalkyl carbamate crosslinking agents as obtained by, for example, reacting polyprimary amines with **ethylene** **carbonate**. The amounts of crosslinking agents used depend on the type and number of groups, in the synthetic resin and crosslinking agent, which react with one another, and on the crosslinking density desired. The weight ratio of synthetic resin to crosslinking agent is in general from 1:9 to 9:1, preferably from 1:1 to 9:1, in particular from 1.5:1 to 4:1.

SUMMARY:

BSUM(51)

It is not absolutely necessary to use separate crosslinking agents since crosslinking functions can also be incorporated into the novel synthetic resin, making it self-crosslinking. This can be done, for example, by a procedure in which the resin obtained after the reaction of (A), (B) and, where relevant, (C) is reacted with a partially blocked polyisocyanate which still contains on average about one free isocyanate group in the molecule. Examples of suitable blocking agents are alcohols, phenols and **oximes**. Another possible method of modifying the novel synthetic resin to render it self-crosslinking comprises introducing .beta.-hydroxyalkyl carbamate groups. To do this, the ketone is first eliminated hydrolytically from the resin according to the invention. The resulting primary amino groups are then converted to .beta.-hydroxyethyl carbamates, for example with **ethylene** **carbonate**.

US PAT NO: 4,575,538 [IMAGE AVAILABLE]

L5: 23 of 29

ABSTRACT:

A process for producing a novel-highly active lanthanide containing catalysts comprising products formed by reacting a lanthanide halide, and an electron donor ligand with an organometal cocatalyst component and its use in the polymerization of olefins, especially olefins such as ethylene, 1,3-butadiene, isoprene and the like. In one embodiment, an organic base is used to increase catalyst activity in those instances where rare earth metal halide-ligand complex is formed with a ligand containing an acidic proton. In another embodiment, diolefins and vinyl aromatics are polymerized in a two-stage process employing a lanthanide complex-organometal cocatalyst in the first stage and a free radical initiator in the second stage.

US PAT NO: 4,575,538 [IMAGE AVAILABLE]

L5: 23 of 29

DATE ISSUED: Mar. 11, 1986

TITLE: Olefin polymerization

INVENTOR: Henry L. Hsieh, Bartlesville, OK
Gene H. C. Yeh, Bartlesville, OK
ASSIGNEE: Phillips Petroleum Company, Bartlesville, OK (U.S. corp.)
APPL-NO: 06/684,407
DATE FILED: Dec. 20, 1984
ART-UNIT: 116
PRIM-EXMR: Patrick P. Garvin
LEGAL-REP: Howard D. Doescher

SUMMARY:

BSUM(24)

Alkyl or aromatic esters of linear or cyclic carboxylic acids having from 1 to about 20 carbon atoms and carbonic acid. Examples include methyl formate, ethyl octonate, n-butyl-eicosanoate, methyl anisate, ethyl benzoate, methyl-para-toluate, diethyl carbonate, methyl ethyl carbonate, di-(3-methylbutyl)carbonate, **ethylene** **carbonate**, diphenyl carbonate, and the like and mixtures.

SUMMARY:

BSUM(26)

Linear and cyclic **oximes** or related oxygen-and nitrogen-containing organic compounds containing from 2 to about 20 carbon atoms such as acetoxime, methyl ethyl ketoxime, caprylaldoxime, ethyl-n-heptadecyl **oxime**, trans-benzaldoxime, benzophenone **oxime**, cyclohexanone **oxime**, 2-octanone **oxime**, pyridine-N-oxide, N,N-diethylhydroxylamine, N-phenylhydroxylamine, N-hydroxypiperidine, and the like and mixtures.

US PAT NO: 4,557,814 [IMAGE AVAILABLE]

L5: 24 of 29

ABSTRACT:

A synthetic resin which carries basic nitrogen groups and can be diluted with water as a result of protonation with an acid can be obtained by reacting an epoxy resin, having a mean molecular weight $M_{sub.n}$ of from 300 to 6,000 and containing on average from 1.5 to 3.0 epoxide groups per molecule, with a diketimine of a primary diamine and, if appropriate, a secondary amine, which may additionally contain a tertiary amino group, a ketimine of a primary monoamine and/or a ketimine of a primary/tertiary diamine.

These resins are useful as binders for coating agents, in particular for the cathodic electrocoating of electrically conductive substrates.

US PAT NO: 4,557,814 [IMAGE AVAILABLE] L5: 24 of 29
DATE ISSUED: Dec. 10, 1985
TITLE: Synthetic resin carrying basic nitrogen groups, and its
preparation
INVENTOR: Eberhard Schupp, Schwetzingen, Federal Republic of Germany
Werner Loch, Erpolzheim, Federal Republic of Germany
Rolf Osterloh, Gruenstadt, Federal Republic of Germany
Klaas Ahlers, Muenster, Federal Republic of Germany
ASSIGNEE: BASF Aktiengesellschaft, Federal Republic of Germany
(foreign corp.)
APPL-NO: 06/629,762
DATE FILED: Jul. 11, 1984
ART-UNIT: 153
PRIM-EXMR: Earl Nielsen
LEGAL-REP: Keil & Weinkauff

SUMMARY:

BSUM(33)

Examples of suitable crosslinking agents for the novel binders are amino resins, such as urea-formaldehyde resins, melamine resins or benzoguanamine resins, phenoplast resins, blocked isocyanate crosslinking agents, crosslinking agents which cure via ester aminolysis and/or transesterification and have on average two or more activated ester groups per molecule, for example .beta.-hydroxyalkyl ester crosslinking agents as described in EP 00 40 867 and carbalkoxymethyl ester crosslinking agents as described in German Patent Application P 32 33 139.8, urea condensates as obtained from, for example, primary di- and/or polyamines, secondary monoamines and urea in the presence or absence of a polyalcohol, e.g. trimethylolpropane, and .beta.-hydroxyalkyl carbamate crosslinking agents as obtained by, for example, reacting a primary polyamine with **ethylene** **carbonate**. The amounts of crosslinking agents used depend on the type and number of groups which react with one another in the synthetic resin and crosslinking agent, and on the degree of crosslinking desired. In general, the weight ratio of the synthetic resin to the crosslinking agent is from 1:9 to 9:1, preferably from 1:1 to 9:1, in particular from 1.5:1 to 4:1.

SUMMARY:

BSUM(34)

It is not absolutely necessary to add separate crosslinking agents, since crosslinking functions can also be incorporated in the novel synthetic resin to make it self-crosslinking. This can be achieved if, for example, the resin obtained from the reaction of (A), (B) and, where

relevant, (C) is reacted with a partially blocked polyisocyanate which also contains on average about one free isocyanate group per molecule. Examples of suitable blocking agents in this case are alcohols, phenols and ****oximes****. Another possible method of modifying the novel synthetic resin to make it self-crosslinking is to introduce .beta.-hydroxyalkyl carbamate groups. To do this, the ketone is first eliminated hydrolytically from the novel resin, and the resulting primary amino groups are then converted to .beta.-hydroxyethyl carbamates, for example with ****ethylene** carbonate****.

US PAT NO: 4,517,359 [IMAGE AVAILABLE]

L5: 25 of 29

ABSTRACT:

11-Methyl-11-aza-4-0-cladinosyl-6-0-desosaminyl-15-ethyl-7,13,14-trihydroxy-3,5,7,9,12,14-hexamethyl-oxacyclopentadecane-2-one and derivatives thereof, such as the 13,14-carbonate and C.sub.1 -C.sub.3 -alkanoyl derivatives thereof. The compounds exhibit antibacterial activity.

US PAT NO: 4,517,359 [IMAGE AVAILABLE]

L5: 25 of 29

DATE ISSUED: May 14, 1985

TITLE: 11-Methyl-11-aza-4-0-cladinosyl-6-0-desosaminyl-15-ethyl-7,13,14-trihydroxy-3,5,7,9,12,14-hexamethyl-oxacyclopentadecane-2-one and derivatives thereof

INVENTOR: Gabrijela Kobrehel, Zagreb, Yugoslavia
Slobodan Djokic, Zagreb, Yugoslavia

ASSIGNEE: Sour Pliva farmaceutska, kemijska prehrambena i kozmeticka industrija, n.sol.o., Zagreb, Yugoslavia (foreign corp.)

APPL-NO: 06/304,481

DATE FILED: Sep. 22, 1981

ART-UNIT: 123

PRIM-EXMR: Nicky Chan

LEGAL-REP: Pollock, Vande Sande & Priddy

SUMMARY:

BSUM(5)

It has further been known that Beckmann's rearrangement of erythromycin A ****oxime****, followed by the reduction of the obtained product, yields a 15-membered semisynthetic antibiotic of the erythromycin series, i.e. 11-aza-10-deoxo-10-dihydro erythromycin A (German Offenlegungsschrift 30 12 533).

SUMMARY:

BSUM(6)

It has also been known that the reaction of erythromycin A with ****ethylene** carbonate**** yields an 11,12-cyclic carbonate of erythromycin A, which is one of those rare erythromycin derivatives that exhibit an improved antibacterial activity if compared with the starting antibiotic (U.S. Pat. No. 3,417,077; Roczn. Chem. 46, 2212-2217, 1972).

US PAT NO: 4,427,822 [IMAGE AVAILABLE]

L5: 26 of 29

ABSTRACT:

A particulate polymer which has imide groups and may be in gelled state can be produced by reacting a polyisocyanate with a polycarboxylic acid having at least one acid anhydride group, and if necessary together with a polycarboxylic acid other than that mentioned above in the presence of a liquid medium containing at least one non-aqueous organic liquid, and thereby obtaining the particulate polymer having imide groups dispersed in the non-aqueous organic liquid.

US PAT NO: 4,427,822 [IMAGE AVAILABLE]

L5: 26 of 29

DATE ISSUED: Jan. 24, 1984

TITLE: Process for producing particulate polymer having imide groups

INVENTOR: Hiroshi Nishizawa, Kitaibaraki, Japan
Yoshiyuki Mukoyama, Hitachi, Japan
Osamu Hirai, Hitachi, Japan

ASSIGNEE: Hitachi Chemical Co., Ltd., Tokyo, Japan (foreign corp.)

APPL-NO: 06/440,570

DATE FILED: Nov. 10, 1982

ART-UNIT: 143

PRIM-EXMR: Maurice J. Welsh

LEGAL-REP: Antonelli, Terry & Wands

SUMMARY:

BSUM(14)

As examples of non-aqueous organic liquid in which the particulate polymer having imide groups formed in this invention is substantially insoluble, there can be used ketones such as acetone, methyl isobutyl ketone, cyclohexanone and the like; esters such as ethyl acetate, butyl acetate and the like; Cellosolves such as methyl Cellosolve acetate, ethyl Cellosolve acetate and the like; ethers such as tetrahydrofuran, dioxane and the like; aliphatic and alicyclic hydrocarbons such as n-hexane, octane, dodecane, ISOPAR-E, ISOPAR-H, ISOPAR-K (trade names of petroleum type saturated aliphatic and alicyclic hydrocarbons having

boiling points ranging from about 40.degree. to 300.degree. C., manufactured by Esso Standard Oil Co.) and the like; aromatic hydrocarbons such as benzene, toluene, xylenes, NISSEKI HISOL-100, NISSEKI HISOL-150 (trade names of petroleum type aromatic hydrocarbons having boiling points ranging from about 80.degree. to 300.degree. C., manufactured by Nippon Sekiyu Kagaku K. K.) and the like; acetonitrile; .gamma.-butyrolactone; **ethylene** **carbonate**, **propylene** **carbonate** and the like. Among these liquids, those having a boiling point of 80.degree. C. or higher are preferable from the viewpoint of reaction temperature. Further, those which can solubilize the reactants are preferable for suppressing the side reactions and proceeding the reaction at a relatively low temperature. Further, the non-aqueous organic liquid may be used in a partial combination with a non-aqueous organic liquid in which the particulate polymer having imide groups is soluble, such as N-methylpyrrolidone, dimethylformamide, dimethylacetamide, phenol, cresol and the like. Such a liquid can be used in such an amount that it causes swelling of the particulate polymer and does not cause agglomeration nor solution of the particulate polymer dispersed in the non-aqueous organic liquid. Preferably, this non-aqueous organic liquid in which the particulate polymer having imide groups is soluble has a poor affinity to the non-aqueous organic liquid in which the particulate polymer having imide groups is substantially insoluble. If it is so, the non-aqueous organic liquid in which the particulate polymer having imide group is soluble is effectively distributed into the dispersion phase of particulate polymer, as the result of which it accelerates the polymerization reaction to yield a particulate polymer of high degree of polymerization.

SUMMARY:

BSUM(15)

Examples of the polyisocyanate usable in this invention include aromatic diisocyanates such as tolylene diisocyanate, xylylene diisocyanate, 4,4'-diphenylether diisocyanate, naphthylene-1,5-diisocyanate, 4,4'-diphenylmethane diisocyanate and the like, aliphatic diisocyanates such as ethylene diisocyanate, 1,4-tetramethylene diisocyanate, 1,6-hexamethylene diisocyanate, 1,12-dodecane diisocyanate and the like; alicyclic diisocyanates such as cyclobutene-1,3-diisocyanate, cyclohexane-1,3- and 1,4-diisocyanates, isophorone diisocyanate and the like; polyisocyanates such as triphenylmethane-4,4',4"-triisocyanate, a polyphenylmethyl polyisocyanate, e.g. phosgenated product of aniline-formaldehyde condensate or the like; and isocyanurate ring-containing polyisocyanates produced by trimerizing these polyisocyanates. From the viewpoint of heat resistance, it is preferable to use, among these polyisocyanates, aromatic diisocyanates such as

tolylene diisocyanate, 4,4'-diphenylmethane diisocyanate and the like and isocyanurate ring-containing polyisocyanates obtainable by the trimerization reaction of these aromatic diisocyanates. The process for producing preferable isocyanurate ring-containing polyisocyanates is mentioned in Japanese Patent Application No. 148,820/78. Said isocyanurate ring-containing polyisocyanates are used as branching component, and their isocyanurate ring skeleton gives excellent heat resistance. For synthesizing a particulate polymer having imide groups which is substantially linear and thermoplastic, bifunctional polyisocyanates are used. For synthesizing a particulate polymer having imide groups which is branched and thermosetting, and for synthesizing a particulate polymer having imide groups which has a three-dimensional network structure and is insoluble in a solvent (gel particle), 3- or higher-functional polyisocyanates are used. These polyisocyanates are used either alone or as a mixture thereof, depending on purposes. In order to control reaction rate in the course of polycondensation reaction and to obtain a stable particle, it is also possible to use said polyisocyanates in the form of being stabilized with an appropriate blocking agent having one active hydrogen in the molecule, such as methanol, n-butanol, benzyl alcohol, .epsilon.-caprolactam, methyl ethyl ketone **oxime**, phenol, cresol or the like.

US PAT NO: 4,328,334 [IMAGE AVAILABLE]

L5: 27 of 29

ABSTRACT:

11-aza-10-deoxo-10-dihydroerythromycin A and derivatives thereof, and process for preparation thereof.

US PAT NO: 4,328,334 [IMAGE AVAILABLE]

L5: 27 of 29

DATE ISSUED: May 4, 1982

TITLE: 11-Aza-10-deoxo-10-dihydroerythromycin A and derivatives thereof as well as a process for their preparation

INVENTOR: Gabrijela Kobrehel, Zagreb, Yugoslavia
Gordana Radobolja, Zagreb, Yugoslavia
Zrinka Tamburasev, Zagreb, Yugoslavia
Slobodan Djokic, Zagreb, Yugoslavia

ASSIGNEE: PLIVA Pharmaceutical and Chemical Works, Zagreb, Yugoslavia (foreign corp.)

APPL-NO: 06/134,816

DATE FILED: Mar. 28, 1980

ART-UNIT: 125

PRIM-EXMR: Johnnie R. Brown

LEGAL-REP: Pollock, Vande Sande & Priddy

SUMMARY:

BSUM(20)

The reduction with complex metal hydrides, e.g. sodium borohydride, is carried out by gradually adding solid NaBH₄ (over about 4 hours) at 4.degree. C. to a methanolic solution of the crude product, obtained by means of Beckmann rearrangement of erythromycin A **oxime**, and thereafter crude 11-aza-10-deoxo-10-dihydroerythromycin A is isolated by conventional methods. The obtained precipitate is suspended in ether, stirred for about 2 hours under ice-cooling, filtered and the filtrate is evaporated as chromatographically (dimethyl formamide: methanol 3:1) pure compound I.

SUMMARY:

BSUM(22)

By the reaction of the compound I with **ethylene carbonate** in the presence of K₂CO₃ in toluene, ethyl acetate or some other inert solvent, 11-aza-10-deoxo-10-dihydroerythromycin A cyclic 13,14-carbonate (VI) is obtained, which by acylation with acid anhydrides of the formula R_ICO--O--COR_{II}, wherein R_I and R_{II} have the meanings as defined above, gives the corresponding acyl derivatives (VII).

SUMMARY:

BSUM(24)

In order to examine the antibacterial action, some new compounds were tested in vitro on a series of gram-positive and gram-negative micro-organisms. The results are shown in Table 1 as minimum inhibitory concentrations (MIC) in mcg/ml in comparision with erythromycin A **oxime**.

SUMMARY:

BSUM(25)

TABLE 1

Test								
bacterium strain		E	EO	I	III	IV	VI	VIII
Streptococcus								
faecalis ATCC 8043		0.05	0.05	0.5	125	175	0.05	

Staphylococcus epidermidis ATCC 12228	0.1	0.1	0.5	200	200	2.5	100
Staphylococcus au- reus ATCC 6538-P	0.5	0.5	0.5	150	200	1.0	100
Micrococcus flavus ATCC 10240	0.05	0.1	0.05	50	175	0.5	20
Sarcina lutea ATCC 9341	0.05	0.05	0.05	10	10	0.1	50
Bacillus cereus var. mycoides ATCC 11778	0.1	0.1	0.5	200	175	2.5	50
Bacillus subtilis ATCC 6633	0.05	0.1	5	175	200	0.5	100
Corynebacterium xerosis NCTC 9755	0.1	0.1	1.0	--	--	--	--
Brucella bronchi- septica ATCC 4617	1.0	1.0	1.0	--	--	--	--
Pseudomonas aeru- ginosa NCTC 10490	50	50	50	--	--	--	--
Klebsiella pneu- moniae ATCC 10031	5.0	5.0	10	--	--	--	--
Escherichia coli ATCC 10536	25	25	10	--	--	--	--
Shigella flexneri II-1819/C	25	50	10	--	--	--	--
Salmonella panama	50	50	50	--	--	--	--

E erythromycin A

EO erythromycin A **oxime**

-- not determined

Roman numbers refer to novel compounds from Examples.

US PAT NO: 4,250,203 [IMAGE AVAILABLE]

L5: 28 of 29

ABSTRACT:

Photopolymerizable compositions using sulfonium salt photoinitiators to effect polymerization of cationically polymerizable monomers as essentially the sole polymerizable material additionally include an organic sulfur compound scavenger to reduce or eliminate the sulfur odor

generated upon photodecomposition of the sulfonium salt photoinitiator. The scavengers are simultaneously activated by the electromagnetic radiation to react with the organic sulfur compound concurrently generated and are selected from the group consisting of aromatic ketones, benzoin compounds, aryloin oxime compounds, organic peroxide compounds, and benzil compounds.

US PAT NO: 4,250,203 [IMAGE AVAILABLE] L5: 28 of 29
DATE ISSUED: Feb. 10, 1981
TITLE: Cationically polymerizable compositions containing
sulfonium salt photoinitiators and odor suppressants and
method of polymerization using same
INVENTOR: Sheldon I. Schlesinger, Windsor, NJ
Dennis E. Kester, Newtown, CT
ASSIGNEE: American Can Company, Greenwich, CT (U.S. corp.)
APPL-NO: 06/071,283
DATE FILED: Aug. 30, 1979
ART-UNIT: 162
PRIM-EXMR: Michael R. Lusignan
LEGAL-REP: Robert P. Auber, Stuart S. Bowie, Peter L. Costas

DETDESC:

DETD(79)

To 16.55 gram portions of this formulation were added a 50 percent by weight solution of impure bis-[4-diphenylsulfonio) phenyl] sulfide bishexafluorophosphate in **propylene** **carbonate** as the photoinitiator and different organic compounds to test their efficacy as sulfide scavengers. These compositions were then coated onto aluminum test panels with a No. 3 wire wound rod and cured under a 360 watt UVIARC lamp (60 watts/inch output). The exposure times indicate the time required to cure the coating to a tack-free condition.

DETDESC:

DETD(80)

Catalyst	Cure
Solution, gm.	
Additive Amount, gm.	
	Time, Sec.
	Odor

0.666	none	--	3	strong mercaptan odor
0.659	.alpha.benzoin	**oxime**		
	0.424	5		some reduction in mercaptan odor
0.666	benzoyl peroxide			
	0.763	3		no mercaptan odor; sweet odor
0.444	xanthene-9-one			
	0.274	3		reduction of mercaptan odor
0.666	thioxanthone			
	0.167	3		slight reduction of mercaptan odor
0.666	2-chlorothio-			
	0.259	3		some reduction of mercaptan odor
	xanthone			
0.666	2,2-azobis-			
	0.516	3		no noticeable reduction in mercaptan odor
	isobutyronitrile			

DETDESC:

DETD(82)

To test the effect upon physical properties, the coating formulation of Example Five was prepared in three aliquots of 16.55 grams to each of which were added 0.666 grams of bis-[4-diphenylsulfonio) phenyl] sulfice bishexafluorophosphate in **propylene** **carbonate** (a 50% solution). To one of these was added 0.167 gram benzoin; and to another was added 1.00 gram benzoin. All coatings of the compositions on aluminum panels exposed to ultraviolet radiation for only one second showed a fingerprint immediately after exposure; the coating without benzoin cured print-free after one second whereas the benzoin-containing coatings required 2-3 seconds. All coatings were immediately print-free when exposed to ultraviolet radiation for 3 seconds.

US PAT NO: 4,199,021 [IMAGE AVAILABLE]

L5: 29 of 29

ABSTRACT:

A thermal energy storage apparatus and method employs a container formed of soda lime glass and having a smooth, defect-free inner wall. The container is filled substantially with a material that can be supercooled to a temperature greater than 5.degree. F., such as **ethylene**

****carbonate****, benzophenone, phenyl sulfoxide, Di-2-pyridyl ketone, phenyl ether, diphenylmethane, ethylene trithiocarbonate, diphenyl carbonate, diphenylamine, 2-benzoylpyridine, 3-benzoylpyridine, 4-benzoylpyridine, 4-methylbenzophenone, 4-bromobenzophenone, phenyl salicylate, diphenylcyclopropanone, benzyl sulfoxide, 4-methoxy-4PR-methylbenzophenone, N-benzoylpiperidine, 3,3PR,4,4PR,5 pentamethoxybenzophenone, 4,4'-Bis-(dimethylamino)-benzophenone, diphenylboron bromide, benzaldehyde, benzophenone ****oxime****, azobenzene. A nucleating means such as a seed crystal, a cold finger or pointed member is movable into the supercoolable material. A heating element heats the supercoolable material above the melting temperature to store heat. The material is then allowed to cool to a supercooled temperature below the melting temperature, but above the natural, spontaneous nucleating temperature. The liquid in each container is selectively initiated into nucleation to release the heat of fusion. The heat may be transferred directly or through a heat exchange unit within the material.

US PAT NO:	4,199,021 [IMAGE AVAILABLE]	L5: 29 of 29
DATE ISSUED:	Apr. 22, 1980	
TITLE:	Thermal energy storage apparatus	
INVENTOR:	Paul E. Thoma, Burlington, WI	
ASSIGNEE:	Johnson Controls, Inc., Milwaukee, WI (U.S. corp.)	
APPL-NO:	05/744,695	
DATE FILED:	Nov. 24, 1976	
ART-UNIT:	345	
PRIM-EXMR:	Albert W. Davis, Jr.	
LEGAL-REP:	Andrus, Scales, Starke & Sawall	

ABSTRACT:

A thermal energy storage apparatus and method employs a container formed of soda lime glass and having a smooth, defect-free inner wall. The container is filled substantially with a material that can be supercooled to a temperature greater than 5.degree. F., such as ****ethylene**** ****carbonate****, benzophenone, phenyl sulfoxide, Di-2-pyridyl ketone, phenyl ether, diphenylmethane, ethylene trithiocarbonate, diphenyl carbonate, diphenylamine, 2-benzoylpyridine, 3-benzoylpyridine, 4-benzoylpyridine, 4-methylbenzophenone, 4-bromobenzophenone, phenyl salicylate, diphenylcyclopropanone, benzyl sulfoxide, 4-methoxy-4PR-methylbenzophenone, N-benzoylpiperidine, 3,3PR,4,4PR,5 pentamethoxybenzophenone, 4,4'-Bis-(dimethylamino)-benzophenone, diphenylboron bromide, benzaldehyde, benzophenone ****oxime****, azobenzene. A nucleating means such as a seed crystal, a cold finger or pointed member is movable into the supercoolable material. A heating element heats the supercoolable material above the melting temperature to store heat. The material is then allowed to cool to a supercooled

temperature below the melting temperature, but above the natural, spontaneous nucleating temperature. The liquid in each container is selectively initiated into nucleation to release the heat of fusion. The heat may be transferred directly or through a heat exchange unit within the material.

SUMMARY:

BSUM(10)

Although any supercoolable material can be employed, available materials which have characteristics particularly adapted to this present invention include **ethylene** **carbonate**, benzophenone, phenyl sulfoxide, Di-2-pyridyl ketone, phenyl ether, diphenylmethane, ethylene trithiocarbonate, diphenyl carbonate, diphenylamine, 2-benzoylpyridine, 3-benzoylpyridine, 4-benzoylpyridine, 4-methylbenzophenone, 4-bromobenzophenone, phenyl salicylate, diphenylcyclopropenone, benzyl sulfoxide, 4-methoxy-4pr-methylbenzophenone, N-benzoylpiperidine, 3,3pr,4,4pr,5 pentamethoxybenzophenone, 4,4'-Bis-(dimethylamino)-benzophenone, diphenylboron bromide, benzalpthalide, benzophenone **oxime**, azobenzene and the like with greater than 5.degree. F. supercooling. Mixtures of such materials with each other and with other materials which form a eutectic composition will also provide supercoolable character. The materials generally operated at elevated temperatures and thus are conveniently stored in natural environments. For example, **ethylene** **carbonate** spontaneously solidifies at approximately 45.degree. F., ethylene trithiocarbonate solidifies at approximately 50.degree. F., and benzophenone solidifies at a temperature below -20.degree. F. These materials are available commercially and the ambient storage temperature is readily held above such a spontaneous solidification temperature for storage. Phenyl salicylate similarly functions with a somewhat longer period of freezing and, therefore, timed release of thermal energy.

DETDESC:

DETD(12)

In the illustrated embodiment, the container 8 is shown as a cylindrical tank having a removable cover 13, through which the conduit 11 passes. In a practical system, the container 8 may be formed of a soda lime glass, which is readily formed as a smooth and essentially imperfection free container and which will not react with the supercoolable material. For example, particularly satisfactory supercoolable materials include benzophenone, 4-benzoylpyridine and **ethylene** **carbonate** for application of normal environmental conditions. **Ethylene** **carbonate**, for example, has a convenient melting temperature of

96.degree. F. and can be readily supercooled to a temperature of around 50.degree. F. without solidification. The carbonate also has a relatively high heat of fusion being on the order of 2.40 kilocalories/mole, and has a heat capacity of 40.6 calories/1.degree. C. mole.

DETDESC:

DETD(14)

	Melting		
	Lowest		
	Point Temp.	Temp. Liq.	Difference

1. **Ethylene** **Carbonate**	96	45	51
2. Benzophenone	117	.dwnarw.	
		-20 .uparw.	137
3. Phenyl Sulfoxide	156	44	112
4. D1-2-Pyridyl Ketone	131	-8	139
5. Phenyl Ether	80.6	17	63.6
6. Diphenylmethane	79	24	55
7. Ethylene Trithiocarbonate	93	50	43
8. Diphenyl Carbonate	175	154	21
9. Diphenylamine	129	87	42
10. 2-Benzoylpyridine	111	.dwnarw.	
		-25 .uparw.	136
3-Benzoylpyridine	104	.dwnarw.	
		-25 .uparw.	129
4-Benzolypyridine	160	27	133
4-Methylbenzophenone	135	.dwnarw.	
		-20 .uparw.	155
4-Bromobenzophenone	180	100	80

Phenyl Salicylate	108	.dwnarw.	
		-32	.uparw.
			140
Diphenylcyclopropenone			
	248	220	28
Benzyl Sulfoxide	275	260	15
4-Methoxy-4Pr-Methylbenzophenone			
	196		
N-Benzoylpiperidine			
	122	.dwnarw.	
		-25	.uparw.
			148

20.

3,3Pr,4,4Pr,5			
Pentamethoxybenzophenone			
	247	Turns to Glass	
4,4'-Bis-(Dimethylamino)-			
Benzophenone	348	217	131
Diphenylboron Bromide			
	77	.dwnarw.	
		-33	.uparw.
			100
Benzalpthalide	221	150	71
Benzophenone **Oxime**			
	290	170	120
Azobenzene	156	120	36

CLAIMS:

CLMS (4)

4. In the method of claim 1 wherein said organic material is a single material selected from the group consisting of **ethylene**
 carbonate, benzophenone, 4-benzoylpyridine.

CLAIMS:

CLMS (5)

5. In the method of claim 1 wherein said material is selected from the group consisting of benzophenone, phenyl sulfoxide, Di-2-Pyridyl ketone, phenyl ether, diphenylmethane, ethylene trithiocarbonate, diphenyl carbonate, diphenylamine, 2-benzoylpyridine, 3-benzoylpyridine, 4-benzoylpyridine, 4-methylbenzophenone, 4-bromobenzophenone, phenyl salicylate, diphenylcyclopropenone, benzyl sulfoxide,

...
4-methoxy-4pr-methylbenzophenone, N-benzoylpiperidine, 3,3pr,4,4pr,5
pentamethoxybenzophenone, 4,4'-Bis-(dimethylamino)-benzophenone,
diphenylboron bromide, benzalpthalide, benzophenone **oxime**,
azobenzene.

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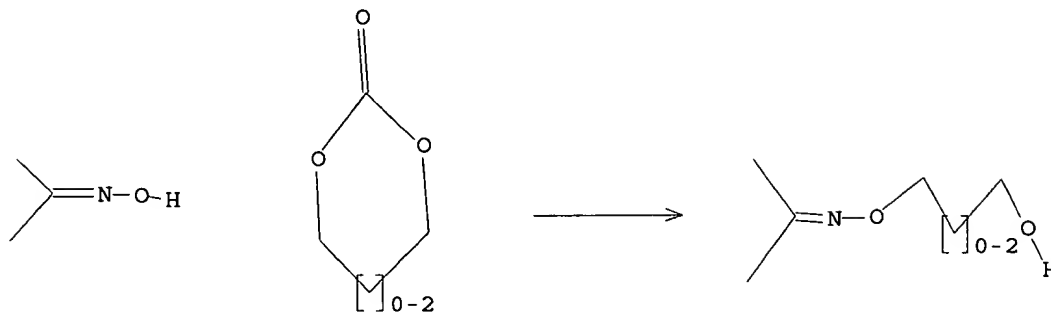
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